ELECTROCHEMICAL STUDY OF REHABILITATION COATINGS IN SOIL SIMULATING CONDITIONS

BY

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أعوذ بالله من الشيطان الرجيم,

بسم الله الرحمن الرحيم,

"فَتَعَالَى اللَّهُ الْمَلِكُ الْحَقُّ ۗ وَلَا تَعْجَلْ بِالْقُرْآنِ مِنْ قَبْلِ أَنْ يُقْضَىٰ إِلَيْكَ وَحْيُهُ ۖ وَقُلْ رَبِّ زِدْنِي عِلْمًا"

سورة طه, ايه, 114

"So high [above all] is Allah, the Sovereign, the Truth. And, [O Muhammad], do not hasten with [recitation of] the Qur'an before its revelation is completed to you, and say, "My Lord, increase me in knowledge.""

SURAH TAHA, verse 114

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LIST OF ABBREVIATIONS

SWCC	:	Saline Water Conversion Corporation
RWTS	:	Riyadh Water Transmission Pipeline
DTRI	:	Desalination Technologies Research Institute
CP	:	Cathodic Protection
EIS	:	Electrochemical Impedance Spectroscopy
PVC	:	Poly vinyl Chloride
ORP	:	Oxidation Reduction Potential
SRB	:	Sulfur Reducing Bacteria
NACE	:	National Association of Corrosion Engineers
ISO	:	International Standard Organization
PE	:	Polyethylene
FBE	:	Fusion Bonded Epoxy
HDPE	:	High Density Polyethylene
HPCC	:	High Performance Composite Coating
MDPE	:	Medium Density Polyethylene
CD	:	Cathodic Disbondment
SCC	:	Stress Corrosion Cracking
SFB	:	Solid Film Backing
LF	:	Low Frequency
FRA	:	Frequency Response Analyzer
RE	:	Reference Electrode
CE	:	Counter Electrode
WE	:	Working Electrode
SCE	:	Standard Calomel Electrode
OES	:	Optical Emission Spectroscopy

CS	:	Carbon Sulfur
FTIR	:	Fourier Transform Infrared Spectroscopy
AC	:	Alternative Current

ABSTRACT (ENGLISH)

Full Name : Ali Bilgaith Al-Sahary

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Saline water conversion corporation (SWCC) operate and maintain water transmission pipelines network in Saudi Arabia estimated as 7175 Km in length with different diameters ranging between 8 and 80 inch. Most of these pipelines are buried in the soil environments and protected externally by coatings and cathodic protection (CP) and using cement mortar lining as an internal protection. Nowadays these assets need rehabilitation in order to maintain integrity and to continue supplying the water demand requirements. However different types of coatings are available in the markets and every coating type has some limitations. Recently viscoelastic or bituminous coatings are used as rehabilitation coatings and the selection of these coatings depends on the degree of corrosiveness of particular environment. International standards such as ISO 21809-3 and NACE SP0109 give some prequalification requirements but different techniques can be used in order to evaluate rehabilitation of coating such as electrochemical impedance spectroscopy (EIS). The main objective of this work was to evaluate four rehabilitation coatings used for underground pipelines based on EIS technique and Gravimetric method. The EIS tests were conducted in soil simulating solution (NS1) at room temperature for more than 8 months with and without outer layer. Meanwhile Distillate water & 1 % NaCl were used in Gravimetric method at two different temperatures (ambient and 30

ºC). In the beginning of EIS test, all the coatings showed outstanding performance due to the nature of high thickness after that the performance of these coatings decreased gradually. However coating B showed better performance even in the absence of outer layer. At the end of exposure of EIS tests, the rehabilitation coatings can be ranked without considering cathodic protection from the best to worst as Coating B with PVC, Coating B without PVC, Coating C with PVC, Coating C without PVC, Coating D with PVC, Coating A and Coating D without PVC. In Gravimetric method, coatings A & D showed the highest water absorption level in all the conditions while coatings B & C obeyed the same behavior in most cases with relatively close structure compounds. Good correlation of the results was observed between the two techniques: EIS & Gravimetric.

ملخص الرسالة ABSTRACT (ARABIC)

الاسم الكامل: علي بلغيث علي السحاري

عنوان الرسالة: دراسة كهروكيميائية لطلاءات إعادة التأهيل في ظروف محاكاة التربة

التخصص: علم مواد وهندسة

تاريخ الدرجة العلمية: مايو 2018

تعمل المؤسسة العامة لتحلية المياة المالحة على تشغيل وصيانة شبكة خطوط أنابيب تقدر بحوالي 7175 كم في المملكة العربية السعودية بأقطار مختلفة تتروح بين 8 و 80 بوصة. معظم خطوط الأنابيب يتم دفنها في التربة ويتم حمايتها خارجيا عن طريق الطلاء والحماية الكاثودية بينما يتم استخدام الملاط الأسمنتي لحمايتها من الداخل. في الوقت الحاضر تحتاج خطوط الأنابيب الى إعادة تأهيل حيث عمرها الذي يبلغ أكثر من 35 سنة وكذلك بغرض مواصلة تلبية الطلب على المياة. لذلك تتوفر أنواع مختلفة من الطلاءات في السوق ولكن يوجد بعض القيود لكل نوع من الأنواع.

حاليا يوجد أحد أنواع الطلاءات يدعى الطلاءات اللزجة أو البتيومينية كطلاءات إعادة تأهيل ويعتمد اختيار هذه الطلاءات على درجة التآكل في بيئة معينة. تحدد المعايير الدولية مثل : الايزو و نيس بعض المتطلبات العامة في اختيار طلاء إعادة التأهيل. بينما توجد بعض التقنيات الأخرى التي تستخدم في تقييم الطلاءات على سبيل المثال: مطيافية المعاوقة الكهروكيميائية (EIS) .

في هذه الأطروحة كان الهدف الأساسي تقييم أربعة أنواع من الطلاءات التي من الممكن استخدامها بغرض إعادة تأهيل خطوط الأنابيب المدفونة بناءا على نتائج المعاوقة الكهروكيميائية والطريق الوزنية لتحديد امتصاص الماء. على الرغم من أنه لم يكن هناك في الدراسات السابقة أي دراسة لرصد تدهور هذه الطلاءات على أساس تقنية المعاوقة الكهروكيمائية.تمت التجارب في محلول محاكاة التربة (NS1) في درجة الحرارة العادية لأكثر من ثمانئة أشهر مع وبدون الطبقة الخارجية المسؤولة عن الحماية الميكانيكية بينما تم استخدام محلولين مختلفين في الطريقة الوزنية (الماء المقطر, 1% NaCl) عند درجات حرارة مختلفة (العادية, 30 م°).

في بداية الغمر في المعاوقة الكهروكيميائية كانت جميع الطلاءات تظهر بأداء متميز بسبب طبيعة سمكها العالي, بعد ذلك انخفض أداءها تدريجيا . كان طلاء ب يظهر أداء أفضل بوجود وفي غياب الطبقة الخارجية. في نهاية الغمر يمكن تصنيف طبقات إعادة التأهيل من دون الأخذ بعين الاعتبار الحماية الكاثودية من الأفضل إلى الأسوأ : طلاء ب , طلاء ب بدون الطبقة الخاجية طلاء ج , طلاء ج بدون الطبقة الخارجية , طلاء د مع الطبفة الخارجية , طلاء أ و طلاء ج بدون الطبقة الخارجية.

في تجربة الاختبارات الوزنية, أظهرت طلاءات أ و د أعلى مستوى امتصاص الماء في معظم الظروف في حين طلاءات ب و ج تمتثل لنفس السلوك في معظم الحالات مع تركيبة قريبة نسبيا.

CHAPTER 1

INTRODUCTION

Saline water conversion corporation (SWCC) operate and maintain a total water transmission pipelines network in Saudi Arabia estimated as 7175 Km in length with different diameters ranging between 8 and 80 inch. Most of them are buried in different corrosive soil conditions employing external coating associated with cathodic protection (CP) as per international standard recommendations. This important asset transports drinking water to the growing population which plays an important role in the development of the Kingdom. Most of existing pipelines are over 30 years in service hence an increase in the failure number raises the subject of rehabilitation approaching design life estimated to be 50 years. Increase of failure rates imply the necessity of life assessment followed by rehabilitation especially for the external surface. Practically, the assessments of buried pipelines condition remain a challenge due to the limitation and high cost of most of used technologies. Thus, investment is more oriented to the rehabilitation projects based on CP readings indicating external coating deterioration.

In parallel, a significant progress has been observed in terms of development, selection criteria and corrosion soil mechanism understanding. External coatings are considered the first line of defense against corrosion for underground pipelines-where they are subjected to more corrosive environments than the above-ground structures and must be able to resist different environment. It is well established fact that there is no perfect coating available in the national and international markets which protect entirely the underground pipelines especially in Saudi Arabian environments. Although, a variety of coatings exist with their advantages and disadvantages that must be considered while selecting a coating.

1.1 Underground Pipeline External Corrosion

Corrosion is an interaction of the material with its surrounding environments, pipelines are submerged in soil conditions which is complicated phenomena especially in presence of coatings and CP. The coating is the passive protection of pipelines once the coating aged or deteriorated the corrosive species can proceed into substrate easily since most of the recorded external pipelines failure is linked with coating defects. Corrosion prevention is not limited to the combination of design, implementation of the design through well written specifications, inspection and construction practices. Therefore, corrosion prevention for buried structures should start with the definition of critical environment conditions before selecting the coating. In this aspect, there is no universally accepted soil corrosion risk assessment methodology. However, most of corrosion professionals adopted the following soil parameters: soil type, moisture, pH, chlorides content, sulfate content, resistivity, and oxidation reduction potential (ORP), backfill material, sulfides (presence of SRB bacteria), potential interference sources (high voltage lines, neighbor CP system, etc.).

According to NACE SP0169 [1], the desirable characteristics of external coating adopted for underground pipelines are: effective electrical insulator; isolation of the pipe against corrosive medium; obey the standard procedures in order to insure good application to pipe; reduce the number of defects during application; achieve the optimum adhesion to the substrate; resistance to development of holidays with time; care handling, storage and installation; stability of electrical resistivity with time; does not shield cathodic protection and–resistance to harsh environment and thermal degradation; ease of application; nontoxic; compatible with cathodic protection and resistance to biodegradation.

1.2 Underground Pipelines Corrosion Factors:

There are several factors that should be considered very well in case of underground structures. One of them is soil type (loam, clay, sand or silt) and chemistry of the soil (Cl^-, SO_4^-, PH etc.) also identifies the corrosiveness of the soil. So the corrosion potential in moist soil will be more negative compared to sandy soil which has more positive potential resulting in high corrosion rate in moist soil condition. Another factor is oxygen content because the oxygen is different from area to area that can exhibits oxygen concentration cell and availability of bacteria in soil which will accelerate the corrosion process [2].

Tator et al. [3] discussed the variables and constrains during the selection of underground pipeline coating system. The author recommends three important parameters that should be investigated very well in order to select the suitable coating system:

1. Pipeline service environments: soil conductivity, pH, Oxygen availability, soil composition (sand, loam, rock), chloride and salts and chemicals.
2. Coating Applications/Handling/Installation variables.

3. Operational Constraints: temperature of the pipe, pipeline history and any related failure.

1.3 Underground Pipeline Rehabilitation

The decision of pipeline rehabilitation should be taken through comprehensive survey about failure history of assets as well as detailed integrity assessment. However, CP readings demands is another performance indicator for the pipelines when the CP current demand cannot more work economically then decision should be taken whether coating or pipeline replacement. Virtually, extensive efforts need to evaluate the old system as well as failure incidents records. On the same time the selection of suitable coatings will be only for the one which can be applied in particular environments taking into consideration the climate variations.

Riyadh Water Transmission Pipelines (RWTP) are considered one of the most important lines being supplied potable water with a distance of more 500 Km to Riyadh city known for its high population (6.5 million) water demand. Corrosion problems either internally or externally should be under control for continuous operation. There are three lines starting from Al-Jubail desalination plants to Riyadh then to Al-Qasim regions which are almost more than 30 years in service. The pipe material used is low carbon steel grades X52 and X60 using cement-mortar lining as internal protection and sintered polyethylene (PE) coating as an external protection combined with a CP system. According to the pipeline authority, a number of external coating related failures increased significantly during the last 10 years. Then, the necessity of rehabilitation project for the three lines becomes very important to ensure continuous potable water supply. There are four commercial available coating systems designed for rehabilitation and repairs which are proposed.

The aim of thesis work was to evaluate the different rehabilitation coatings performance and ranking them in term of best corrosion protection system that could be used as a good option for Riyadh water transmission pipelines without considering the compatibility with cathodic protection (CP).

CHAPTER 2

LITERTURE REVIEW

2.1 Conventional Rehabilitation Coatings

Coatings known from early centuries providing protection for the substrate, they classified as organic and inorganic coatings. Each coating system has its own advantages and disadvantages where some of the coatings cannot be applied in the field due to environmental issues. NACE 0169 [4] classified the generic external coatings based on the substrate whether the coating will applied on carbon steel or cast iron material, Table 2.1 summarizes that classification. Many other authors classify the external underground pipeline coatings as field or plant-applied coatings as shown in Table 2.2 [5, 6]. Mamish [7] described pipeline coatings as main line and girth weld coatings, Main line includes: Cold Applied tapes, Fused tapes , Fusion Bonded Epoxy (FBE), 2 part Urethane and 2&3 layer polyethylene while girth weld includes: Shrink Sleeves , Cold Applied tapes and 2 part liquid epoxy.

Table 2. 1 Generic External Coating Systems applied on Underground Pipelines as per NACE 0169.

Underground Pipe Material	Carbon Steel Pipe	Ductile Iron Pipe
External Coating Type	Asphalt/Coal Tar Enamel + Concrete	Adhesive Tape
	Coal Tar Enamel	Extruded Polyethylene
	Cold-Applied & Hot-Applied Tape	Reinforced Cement Mortar
	Concrete	Field Joint Coatings
	Elastomeric Materials (Polychloroprene or equivalent)	Mastic Coatings
	Field-Applied Coatings for Repair and Rehabilitation	Polyurethane
	Field Joint Coatings	Polyethylene Sleeving
	Fusion-Bonded Epoxy	Wax
	Fusion-Bonded Epoxy + Concrete	Zinc Coatings
	Liquid Epoxy	
	Mastic Coatings	
	Multilayer Epoxy Polyethylene	
	Multilayer (including FBE primer) Polyethylene (PE) & Polypropylene (PP) Anticorrosion	
	Polyolefin Coatings	
	Polyurethane	
	Prefabricated films	
	Wax	

Table 2. 2 Underground Pipeline External Coatings Types based on application.

Coating Type	Coatings Name	Comments	Ref.
Plant-Applied	Fusion Bonded Epoxy (FBE) , High Density Polyethylene (HDPE), Urethanes …etc.		[5]
	FBE 3-layers(FBE+2-Polyolefine layers) Coal Tar enamel	FBE has no shielding Health concern to stop Coal Enamel	[6]
Field-Applied	Spray Coating (epoxy, urethane, Zn…etc.), residues of refinery (waxes, petrolatum), bitumen based coating and single or multi-layer Polyethylene (PE)/butyl tapes		[5]
	FBE Tape coatings Geotextile (mesh) backed tapes Two part epoxies Shrink Sleeves Coal tar ,urethanes and liquid coatings	FBE can be applied in girth welds only. Polyolefin or polyvinyl chloride (PVC) with a bituminous or butyl compound & Shrink sleeves causing (CP shielding) No shielding in mesh tapes	[6]

As described above several external coatings systems could be used for underground pipelines to isolate them against corrosive media combined with CP system. However, some of these coatings could be incompatible with CP or cannot be suggested for rehabilitation project due to the difficulty to be applied in field conditions. In the following brief description of general underground pipeline coatings:

2.1.1 Coal Tar Enamel

It is a polymeric coating produced from the plasticization of coal tar pitch, coal and distillate. The system consists of a primer, coal tar, glass fiber inner wrap and glass fiber outer wrap. This system has many advantages such as the good adhesion to the substrate without shielding CP current. However, nowadays it has been stopped due to health concerns as well as little manufacturer.

2.1.2 Fusion Bonded Epoxy (FBE)

It is a thermosetting materials applied in its powder form on the surface of the steel then subject to heat source to achieve the final dry coating film. FBE have a good history of use due to its good adhesion and low cathodic current demand. However, its drawback is on its application which require high temperature (energy consumption) raising its cost. The dual layer configuration is usually suggested for better mechanical resistance.

2.1.3 Two-layers polyolefin coatings

The primary coat is butyl –based adhesive or asphalt –based mastics and the outer layer is a side-extruded polyolefin layer (HDPE[1]). The advantages of this product are easy application and good bonding to the substrate but the disadvantages is the susceptibility to shield CP current.

2.1.4 Three-layers polyolefin coatings

It is consisting of FBE or liquid epoxy primer and copolymer adhesive intermediate or tie layer top coated with a polyolefin. The advantages and disadvantages are same of two layers.

[1] High Density Polyethylene

2.1.5 Heat Shrink tapes

They consist of a thick, radiation cross-linked polyethylene –based backing coated with high shear strength, thermoplastic, hot-melt adhesive. Table 2.3 summarizes the abovementioned organic coatings characteristics and limitations:

Table 2. 3 Characteristics and Limitations of Pipeline Coating Types [8].

Pipe Coating	Characteristics	Limitations
Coal tar enamels	• Does not shield CP current • The bond strength to the substrate	• Health concerns • No more manufacturer & applicator
Mill-applied tape systems	• Ease of application • Good bond to the steel	• Cathodic disbonding due to soil stress
Crosshead-extruded polyolefin asphalt/butyl adhesive	• Ease of application	• Poor adhesion to steel • Sagging
Dual-side-extruded polyolefin with butyl adhesive	• Good adhesion to steel • Ease of application	• Limited applicators
Fusion-bonded	• Excellent resistance to cathodic disbondment • Good bond to steel	• high temperature required • moisture absorption
Multi-layer epoxy/ extruded polyolefin systems	• Excellent adhesion to steel	• Higher initial cost • Susceptibility to shield CP current

Fu et.al [9] investigated the permeability of the high performance composite coating (HPCC) to CP and compare it to FBE and PE coatings. The authors used a double –cell system through Corrosion potential, potentiostatic polarization current density and EIS Water permeability was measured by Gravimetric method where coating systems ranked from the higher to the lower as FBE > MDPE[2] > HPCC. For the permeability of coating to CP current coatings were ranked as HPCC >FBE> MDPE.

Lee et al. [10] used pulsed Potentiostatic test to evaluate PE and coal-tar enamel coated SS400[3] for buried pipelines with artificial holiday and two different thicknesses. The results were compared to the un-coated Stainless steel and the cycles were repeated 7 times to accelerate the degradation of the coating. The obtained performance results were function of the coating thickness with good corrosion protection for thicker coating. PE was showed less delaminated area and higher performance than coal-tar enamel coating as shown in Figure 2.1.

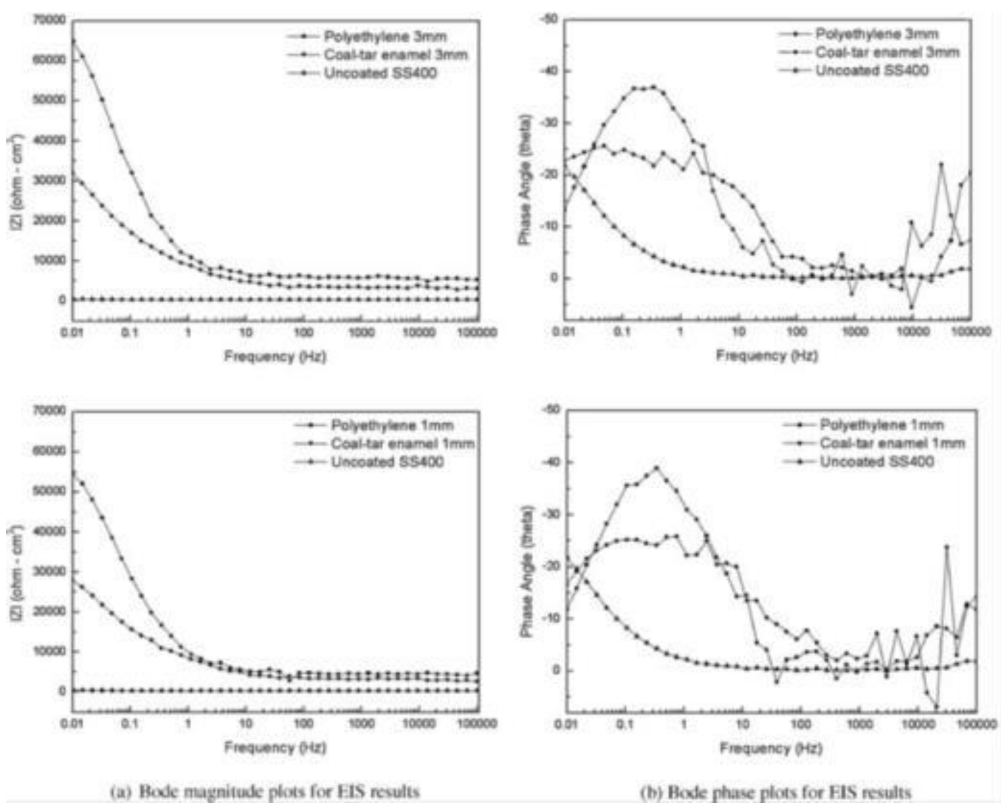

Figure 2. 1 Bode & Phase plots of coated PE and coal tar enamel [10].

[2] Medium density Polyethylene
[3] Martensitic stainless steel

2.2 Rehabilitation Coatings

Rehabilitation Coatings are the radical solution for existing aged underground pipelines in order to eliminate the expected failure due to degradation of old assets and to increase the life of underground pipeline. There is no clear classification of Rehabilitation coatings in the literature while NACE classify field-Applied Bonded Tape Coatings, these coatings can be used as repair or rehabilitation coatings as single or multi-layers. Table.2.4 & Table 2.5 show the advantages and disadvantages of different coatings that could be used in the underground pipelines as rehabilitation, repair and field joints [11, 12, 13, and 14].

Table 2. 4 Advantages and disadvantages of different Rehabilitation Coatings.

Rehabilitation Coating Type	Advantages	Disadvantages	Ref.
100% Solid liquid urethane	Flexibility Single coat Fast curing High abrasion CD resistance at <50°C Tensile adhesion	Moisture Sensitive Poor CD resistance at >50°C Poor hot water adhesion Poor impact resistance at subzero temp.	[12]
100% Solid epoxy/ urethane	CD resistance at <80°C Improved tensile adhesion Improve hot water adhesion	Slower curing than urethane Curing stops at 50°C	[12]
100% Solid epoxy coatings	Improved CD resistance up to 95°C Improved tensile and hot water adhesion Sag resistance	Slower curing than urethane Curing stops at 50°C Increase temperature formulations.	[12]
High temperature epoxy	Cathodic Disbonding (CD) resistance at 150°C		[12]
Low temperature epoxy	cure at 0 °C		[12]
Damp surface epoxy:	CD resistance at 80°C.		[12]

Coal Tar and bitumen based coatings:		Adhesion loss when aged Causing CP current demands increase.	[12]
Two-ply Tape system (PVC and PE tapes):		Removal of Plasticizers leading to adhesion loss.	[13]
Self-amalgamating three-ply Tapes	Stabilizers to avoid spiral corrosion.		[13]
Tapes	Surface Tolerant Easy application	Sensitive to surface preparation Poor adhesion at overlap Not suitable (50-60C) Low resistance to soil stress	[14]

Table 2. 5 Underground Coatings advantages and disadvantages for field joints.

Coating Type	Advantages	Disadvantages
FBE	Formation of magnetite layer. Compatible with CP.	Sensitive to SP
HSS	Fast application. Design Temperature up to 100 °C.	
Polyurethane	Durable & Corrosion Resistance.	High cost. Limited to bends and field joints.
Flame Sprayed PE	Ease of use. Compatible with main pipelines coating.	
Cold Wrapped Tape	Low cost. Easy application.	Low resistance to soil stress. Thermal ageing. Poor adhesion.

Depending on the scope of the coating operation, i.e., new construction or repair/ rehabilitation, end users could adopt either conventional organic coatings or specific type of coatings (tapes) as classified in NACE SP0109 shown in Table 2.6. Tape coating systems are the most suitable choice for rehabilitation because it can be applied whether the pipeline is in or out of service or segmented, it can be single or multilayer systems as it can be seen in Figure.2.2 and is classified as following:

2.2.1 Single coat system

1. Hot Applied systems
2. Cold applied systems

2.2.2 Multilayer coat system

1. Inner layer
2. Mechanical outer layer.

Table 2. 6 Tape System as per NACE SP 0109 [15].

Tape	Description	Application (Cold or Hot)
Bituminous tape	It is a coal tar base or bituminous base coating material supported on a fabric of organic or inorganic fibers (min. thickness 1.3mm)	Hot Applied
Polymeric tapes	It is a compound adhesive to metal and generally bonded to a flexible polymer film and may contain a synthetic reinforcement. These tapes can be Laminate ,Reinforced , and Thin Backing	Cold or Hot Applied
• Laminate Polymer tape	A flexible polymer film coated on one or both sides with an adhesive compound. The adhesive compound may be reinforced with synthetic fibers.(min. thickness 0.89 mm)	Cold or Hot Applied
• Reinforced Polymer tape	A woven mesh, polyolefin fabric bonded to the outside surface of rubberized bituminous or modified solid adhesive types of coating materials (min. thickness 0.89 mm).	Cold Applied
• Thin backing polymeric tape	An adhesive compound.it may contain a flexible polymer film (thickness < 203µm (8mil) to avoid overstretching during application.	Cold Applied

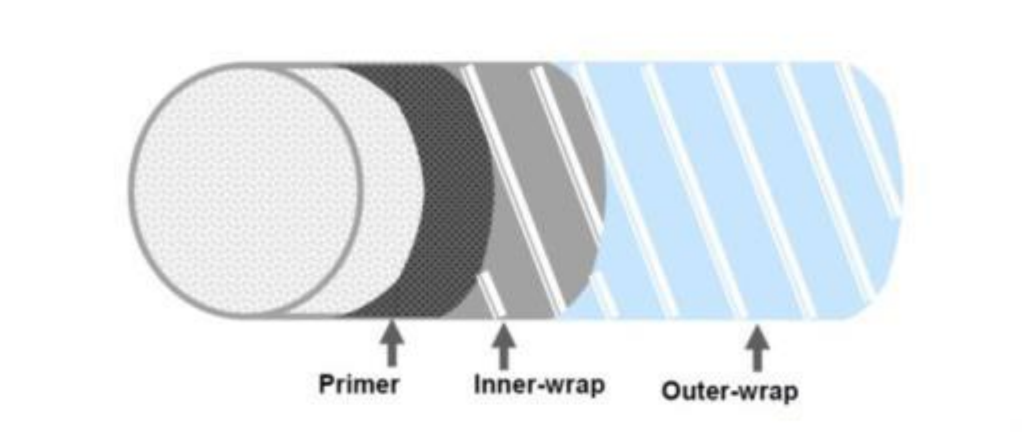

Figure 2. 2 Cold Applied Tape System. (L.Mamish n.d.) [7].

Recently the chance of using non-crystalline low viscosity coatings as rehabilitation coatings as main pipelines as well as girth weld is increasing due to its ease of applications and competitive price. The low viscosity coatings consists of a polar not cross-linked polyolefin these coatings act as fluid with wetting for adhesion improvements purposes , Since there will be only cohesive failure and it can easily heal the irregularities in the substrate and it is an inert and amorphous materials (no reaction) to prevent the pressure build up within the coatings, it will also prevent MIC occurrence because the composition contain an organic polymeric with inorganic filler (No nitrogen) [5].Polymeric or viscos –elastic coatings consists of poly-isobutene which only has covalent bonds contains hydrogen and carbon only having cold flow and non-cross link properties but this type of coatings needs 50 % overlap to strengthen the edges of the materials , there is no need of any chemical reaction to achieve the adhesion, as it adhesive very well on the substrate after application [16] .In one of studies carried by Richard et.al [17] , they discussed coating systems consist of a liquid adhesive covered by a spiral wrapping with geo-textile fabric backed rubberized bituminous compound and non-shielding outer wrap uses primer to fill the surface irregularities and react with bituminous to enhance the adhesion properties to the substrate and he mention that the reported failure related to this product is less than 10 failures and some of them was due to inadequate application and they found water underneath the coating system indicate pH of 10 ,which reflect the effectiveness of CP system (no corrosion),they compare this product with solid film backings SFB (Shrink sleeves and tapes) since the SFB have high dielectric strength they will lead to shielding of CP current and the other problem related to SFB is wrinkling due to soil stress specially in case of improper application. The disadvantages of tape system as per Roland [18] are poor adhesion at overlap especially a butyl rubber ,not suitable to be used in temperature range (50-60 °C) and it is low resistance to soil stress.

According to ISO 20809 the classification of field joint coating as following:

Table 2. 7 Classification of Field Joint Coatings according to ISO 20809 (ISO 2015) [19].

Serial Number	Type of FJC
1	Hot-applied bituminous tape coatings
2	2.1 Petrolatum tape coatings 2.2 wax tape coatings
3	Cold-applied polymeric tape coatings
4	4.1 Non-crystalline low-viscosity polyolefin based coatings with polymeric tape outer wrap 4.2 Non-crystalline low-viscosity polyolefin based coatings with heat shrinkable material outer wrap
5	5.1 Coatings based on heat-shrinkable materials, polyethylene-base, applied without primer 5.2 Coatings based on heat-shrinkable materials, polyethylene-based, applied over a liquid epoxy, fusion-bonded epoxy, or other compatible primer layer 5.3 Coatings based on heat-shrinkable materials, polyolefin-based, with reinforced backing (hybrid),applied over a liquid epoxy, fusion-bonded epoxy, or other compatible primer layer 5.4 Coatings based on heat-shrinkable materials, polypropylene-based, applied over a liquid epoxy, fusion-bonded epoxy, or other compatible primer layer
6	Hot-applied microcrystalline wax coatings
7	7.1 Elastomeric coatings, polychloroprene-based 7.2 Elastomeric coatings, EPDM-based
8	8.1 Single-layer fusion-bonded epoxy powder coatings 8.2 Two-layer fusion-bonded epoxy powder coatings
9	9.1 Liquid applied epoxy based coatings 9.2 Liquid applied polyurethane based coatings 9.3 Fibre-reinforced epoxy based coatings 9.4 Fibre-reinforced vinylester based coatings 9.5 Cast solid polyurethane based coatings
10	10.1 Coatings based on flame-sprayed polypropylene powder applied over an epoxy layer 10.2 Coatings based on polypropylene tapes/sheets hot-applied over an epoxy layer 10.3 Coatings based on injection-moulded polypropylene over an epoxy layer 10.4 Coatings based on flame-sprayed polyethylene powder applied over an epoxy layer 10.5 Coatings based on polyethylene tapes/sheets hot-applied over an epoxy layer
11	Thermal spray aluminum (TSA) coatings

Alliston, *et al.* [12] mentioned about the recent developments of rehabilitation coatings including:

- High temperature epoxy: CD resistance at 150°C
- Low temperature epoxy: cure at 0 °C
- Damp surface epoxy: CD resistance at 80°C.

Schad [13] discusses many coatings system that can be used for rehabilitation projects as follow:

- ✓ Coal Tar and bitumen based coatings: brittle behavior with ageing leading to cracks and defects which will loss the bond with steel substrate. So, in this case the operator will squeeze to increase CP current above economically values to achieve desired protection and this will lead to H_2 formation which induce a Stress Corrosion Cracking (SCC) process.
- ✓ Two-ply Tape system (PVC and PE tapes): These coatings systems have plasticizers that with time they will diffuse out from the PVC resulting in loss of adhesion between coating system and substrate. Plastic compounds (petrolatum and butyl rubber) and rigid compounds (polyurethane epoxy resins) are filling the irregularities in the whole pipe surface and have low permeability.
- ✓ Self-amalgamating three-ply Tapes: they contain carrier film of stabilized polyethylene coated with a butyl rubber on both sides. This layer is self-amalgamate in the overlap area. This had to come to substitute the drawback of two layers system which will have a gap between adhesive layer and carrier film which leads to spiral corrosion.

The visco-elastic coatings can be used as rehabilitation coatings applied to main pipelines as well as girth weld due to their easy applications and competitive price. These coatings consist of a polar not cross-linked polyolefin. They act as fluid with wetting characteristics for adhesion improvements purposes. In case of failure it should be cohesive failure with self-healing of the irregularities in the substrate. In addition, these coatings provide high chemical and biodegradation resistance [5].

Polymeric or viscos–elastic coatings consist of poly-isobutene which have only covalent bonds containing hydrogen and carbon only and also having cold flow and non-cross link properties but this type of coatings need–50 % overlap to strength the edges of the materials and no need any chemical reaction to achieve the adhesion as it directly adhesive very well on the substrate after application [20].

In one of studies carried by Norsworthy *et al.* [21] discussed coating system consisting of a liquid adhesive covered by spiral wrapping with geo-textile fabric backed rubberized bituminous compound and non-shielding outer wrap uses primer to fill the surface irregularities and reacts with bituminous to enhance the adhesion properties to the substrate. He mentions that the reported failure related to this product is less than 10 failures and some of them were due to inadequate application. There was found water underneath the coating system indicating pH of 10 which reflects the effectiveness of CP system (no corrosion), and he compares this product with solid film backings (Shrink sleeves and tapes) since the SFB have high dielectric strength. They will lead to shielding of CP current and the other problems related to SFB are wrinkling due to soil stress especially in case of improper application. The disadvantages of tape system as per *Roland palmer* are poor adhesion at overlap especially a butyl rubber, not suitable to be used in temperature range (50-60 °C) and it is low resistance to soil stress. Also he mentions other types of coatings; the following table summarizes the advantages and disadvantages of each system:

Table 2. 8 Advantages and disadvantages of some rehabilitation Coatings.

Coating System	Advantages	Disadvantages
Tapes	• Surface Tolerant • Easy application	• Sensitive to surface preparation • Poor adhesion at overlap • Not suitable (50-60C) • Low resistance to soil stress
Liquid Epoxy	• Can be used up to 90C • Resistance to soil stress and impact • Low current requirements for CP • Resistance to CD • Good adhesion • Resist holidays	• White metal finish needed • Sensitive to moisture during application • Not cure at low temperature • Needs special equipment for application
Liquid Polyurethane	• Fully cured in few hours • Soil shear resistance and abrasion • Good adhesion • Low current requirements for CP • Resistance to CD	• White metal finish needed • Sensitive to moisture during application • Needs special equipment for application • High thickness required

Polymeric tapes can be classified as cold or hot applied tapes [18]:

Cold applied Tapes:

A blend of butyl rubber and synthetic resin in the form of tape and it is covered with high density polyethylene or polypropylene as outer layer for mechanical protection.

Hot applied Tapes:

They consist of thermally adhesive primer layer, thermoplastic elastomers adhesive layer and thermoplastic outer layer.

Visco-elastic coating (STOPAQ CZ) wrapping band system with 3mm thickness was studied in both laboratory and field conditions. Holiday detection, Impact resistance, Chemical resistance, EIS and Cathodic Disbonding tests were carried out and compared with the field trail and no holidays were found. The impact test results were approved the self-recovering of this type of coating. This type of chemically coating was stable and the solution was not affected or color changed. EIS, the impedance also shows good value without artificial defects but after the defects it becomes low (Fig.4). Visually, no visible damage was found at CD test. The maximum operating temperature for this product was 70 ºC. For the field tests the pipeline was monitored every 12 months for a total of 4 years of exposure and window test was carried out but visually no any visible damage or corrosion was found which approve the results of the laboratory tests [22].

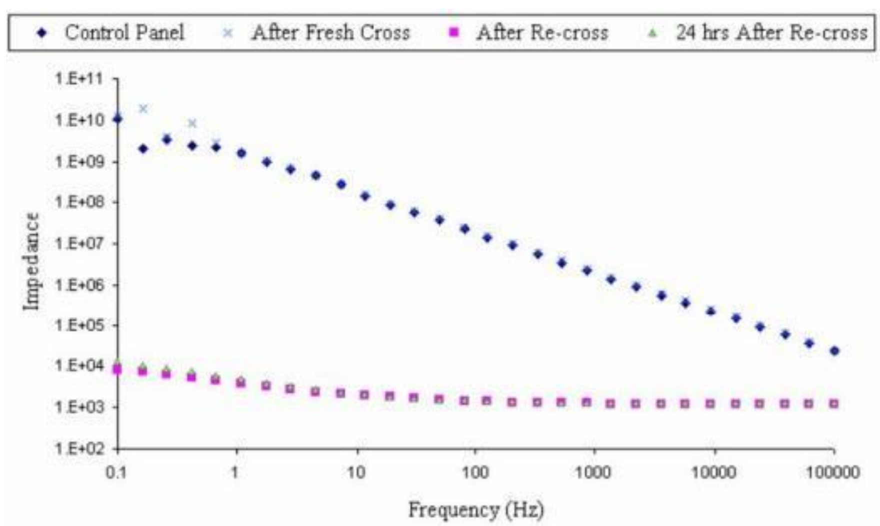

Figure 2. 3 Electrochemical Impedance Results from Coated Panel.

(Romano 2005) [23] Tapes are used for rehabilitation of pipelines either single or multi layers cold applied tape system. It consists of primer and adhesive layer, the primer is to fill the surface irregularities and the adhesive layer which is blended with butyl rubber and synthetic resin to obtain the adhesion of the coating on the substrate and the outer layer of high density polyethylene (HDPE) or polypropylene to resist the mechanical and soil stresses but hot applied tape system is made of a thermally activated primer layer, a thermoplastic adhesive layer and a thermoplastic outer layer of HDPE.

Figure.3.7 shows E_{corr} vs. time for coated steel with different concentration of conductive polymer polyaniline (PAni) as an anticorrosion agent. The monitoring of free corrosion potential along time can give overview of the coating performance in term of corrosion protection (A. F. Baldissera 2010).[24]

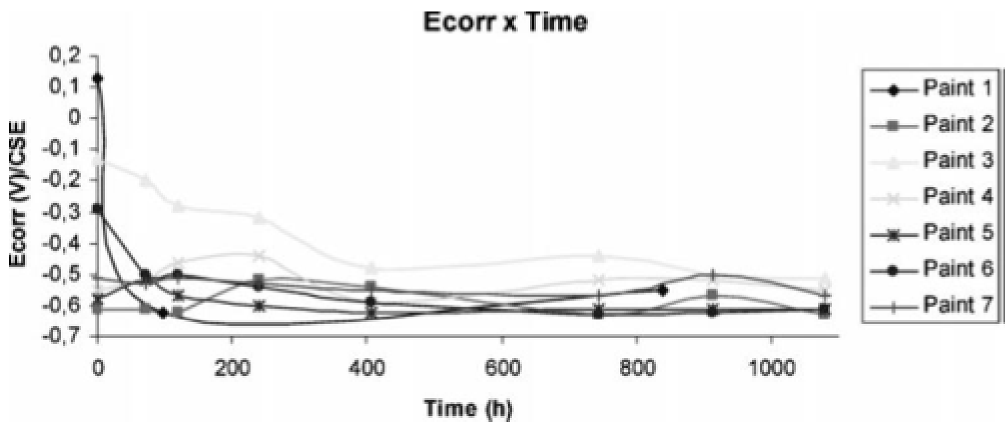

Figure 2. 4 Ecorr vs. time measured in 3.5% NaCl.

Nowadays, visco-elastic polymeric coating tapes are considered the best choices for repair and rehabilitations due to the ease of their application, good barrier properties, self-healing, chemical resistance and economical aspect.

However, limited numbers of studies are available in literatures discussing viscoelastic coatings.

(E. M. Fayyad 2014) [25] Mentioned that self-healing coatings cab be intrinsic or extrinsic, in intrinsic the healing purpose cab be done through hydrogen bonding, monomeric coupling or thermal reactions but in extrinsic can be done through adding components in coating structure.

2.3 Electrochemical Impedance Spectroscopy for Coatings Evaluation

It is called spectroscopy because it is concerned with measuring the impedance versus wide range of frequencies and it can be used to evaluate the coating through monitoring the ingress of ions and other species into coating and to simulate the corrosion behavior through electric circuit for coating / substrate interface system. Actually this type of test can measure the coating impedance which gives indication on degradation of the coating without affecting the failure mechanism as outlined by *Olivier et al.* [26].

Hensley et al. estimated the lifetime prediction of an epoxy polyamide –coated AISI 1010 steel and compared it with an epoxy/chromate conversion–coated magnesium alloy so he focused on several EIS parameters. Following are the most important one:

i. Low frequency (LF) resistance.

ii. Coating resistance.

iii. Breakpoint frequency.

So, he concluded the threshold LF resistance values of 10^7 and 10^8-10^9 Ω-cm^2 for coated steel and coated Mg alloy respectively. They mention that the coating performance can be investigated through LF resistance [27].

Felhosi et al. use EIS technique to study the environmentally-friendly pigments on the organic coatings performance. So they mentioned that from this type of technique especially in Nyquist plot you can extract the dielectric constant of the coatings at just the immersion time or at low frequency values and the properties of coating from the high frequency region but the electrochemical reactions can be getting through the intermediate frequency regions [28].

The following schematic diagram shows the parts included in the electrochemical impedance spectroscopy: Frequency Response Analyzer (FRA) which is the important part in impedance measurements because FRA measure the real and imaginary parts of the impedance. Potentiostat, three electrodes are getting out from potentiostat the reference electrode (RE) e.g. Cu/$CuSo_4^-$ or Standard Calomel Electrode (SCE), Counter electrode (CE) e.g. graphite electrode and working electrode (WE) which is the sample to be tested and finally coupled with computer to monitor and store the data.

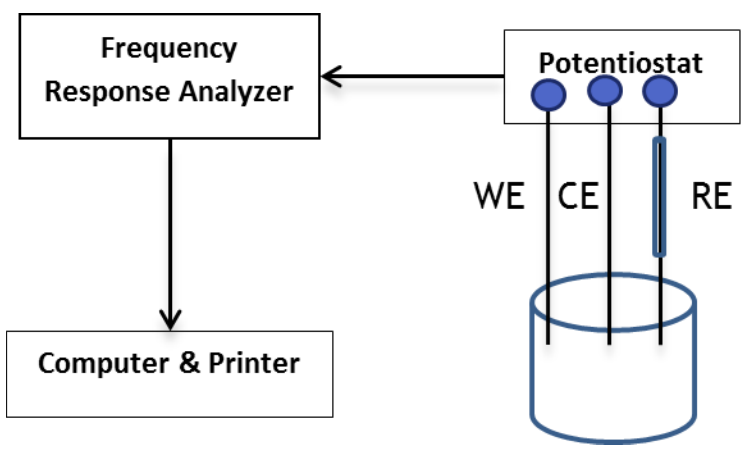

Figure 2. 5 AC Impedance Instrument Components.

Gibitta et al. used EIS to compare the results with field results so he found the EIS technique is valuable and repeatable results comparing with field results and he mentions that the frequency needed to evaluate the coating properties (10^2-10^4 Hz) considered as medium frequency [29].

The following circuit shows the simple electrical circuits that can reflects the corrosion phenomena in this technique (E.McCafferty 2009) [30]:

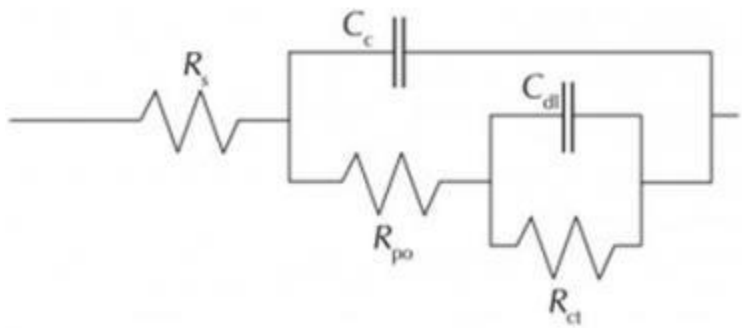

Figure 2. 6 Simple Circuit for Coating Test Simulation.

Where Rs is electrolyte or solution resistance, Cdl is double layer coating capacitance, Rp is polarization resistance, Rcp is pore resistance of the coating and Cc is coating capacitance. So the coating capacitance can be calculated through the following formula:

$$Cc = \frac{\epsilon\epsilon. A}{I}$$

Where ϵ: the dielectric constant of the coating, ϵ.: the dielectric constant of the free space, A: area and I is the thickness of the coating. The system should measure the complex impedance Z (jω) =Z' +Z" in plane plots (Z' vs. Z") or in Bode plots (log |Z|) vs. log ω and phase angle Ø vs. log ω with (|Z|=Z'2+Z"2)1/2, Ø=arc tan (Z"/ Z') and ω=2πf).

So if there is any delamination in the coating then both resistance polarization and pore resistance of the coating will decrease and both capacitance double layer and coating capacitance will increase due to water absorption.

There are a lot of important parameters during electrochemical study; the following are some of these (Philip A.Schweitzer 2006):

- Electrode Potential
- Polarization Characteristics
- Electrical Resistance
- Capacity
- Diffusion Characteristics

Electrode Potential:

When the permeability of the coating is low, then it will impede the ionic motion of the substrate in the presence of a coating defect this will be considered as start point of corrosion because it will play the role of anodic area and the remaining area which is adhered by the coating will be cathodic one and then the potential difference will be started in this case and the potential of defect area will be more negative than cathodic area potential. The following are the steps how this happens:

The anodic reaction will be continued in the defects area (pores, pinhole etc) and the current density in this area is very high then the substrate will become more negative potentials. The interfaces between coating defect (anodic) and substrate or metal (cathodic) continued with shifting the potential to positive side.

Polarization Characteristics

Polarization resistance is an important parameter for judging on the coating if it's permeable of electrochemical process or not.

Electrical Resistance:

The electrical resistance of the coating can be measured via alternating current with variable frequencies and it is classified into active and reactive (polarization) resistance.

The following equation is reflecting the tangent of the angle of the dielectric losses of the coating.

$$\tan\delta = \frac{1}{2\pi fCR}$$

Where f: frequency, C: Capacitance and R: Resistance.

Abrupt change in the tangent of angle of the dielectric losses means that the coating is highly permeable to electrolyte.

Diffusion Characteristics:

The kinetics of the cathodic process of substrate depend on the specious (Water, O_2) diffusion velocity in the coating and vice versa for anodic process depending on the metal ions velocity.

All above data can be plotted using two different methods either Bode or Nyquist plot:

Bode plot: The following plot is Bode plot showing impedance versus Frequencies or phase angle versus frequencies:

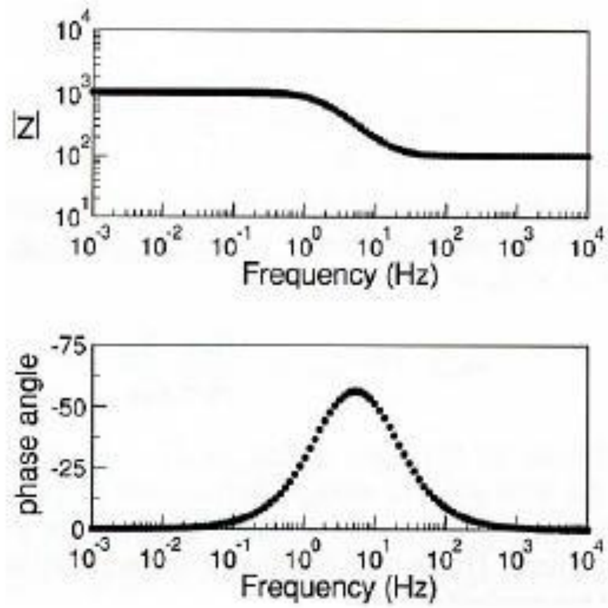

Figure 2. 7 Bode & Phase modulus Plot.

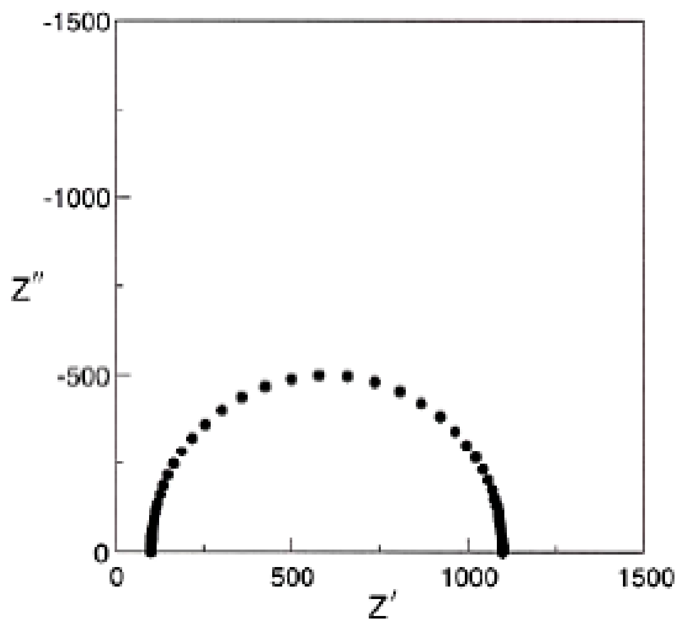

Figure 2. 8 Nyquist Plot.

The above figures show the imaginary impedance versus real impedance for corroding sample so solution and polarization resistances can be extracted from the figure.

Tsai et al. Investigated polybutadine coated steel using EIS technique with different steel surface treatments [31].

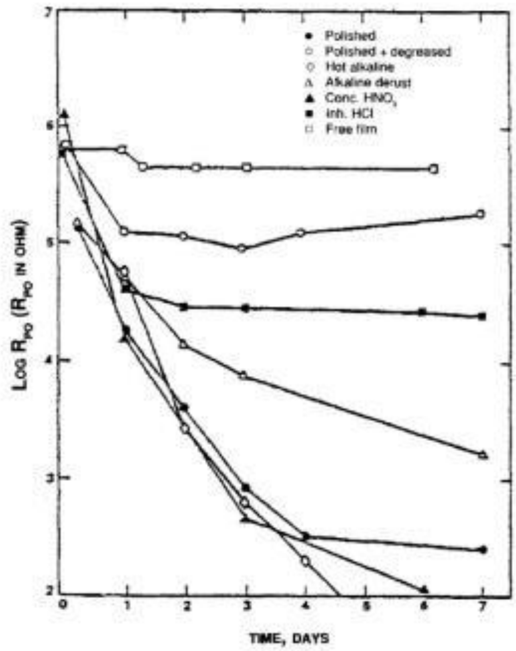

Figure 2. 9 Time dependence of polarization resistance (Tsai 1991).

And then they model this technique by using delamination data according to ASTM 610, the results were agreed with modeling and other parameters that can be obtained from impedance spectra such as breakdown frequency and phase angle to follow the corrosion propagation.

Li et al. they made ranking of different soft coating performance based on EIS technique quantitatively and measured the time of water uptake through diffusion coefficient and coating thickness so they found this technique is useful specially in case of earlier damage of coating through charge transfer resistance parameter [32].

Liu et al. showed the effect of addition of pigments concentration in epoxy coating using EIS technique by following Fick's second law to measure the diffusion coefficients of water. So they found that the diffusion coefficient decreased through increasing pigments concentrations [33].

Bisphenol A and Novalac coatings were tested in 30g/L NaCl at 30,40,60 and 80 °C for 5 months exposure period ,the impedance values at 10 mHz and breakpoint frequency were plotted against time, the impedance was decreased with time specially at elevated temperatures and the breakpoint frequency was increased faster at elevated temperatures in both coatings as a result the water absorption is affecting the coatings properties specially at high temperatures [34].

Polyurethane coating modified by Fe_2O_3 nanoparticles was used on steel substrate in order to compare it with unmodified one so the impedance at low frequency of modified one was very high compare to unmodified coating ,lower breakpoint frequency was noticed in modified coating means better corrosion protection and less delaminated area [35].

CHAPTER 3

MOTIVATION AND OBJECTIVES

3.1 Motivation

The pipelines are very important assets in worldwide industries so the integrity is critical for those structures. The coating is the best method to isolate the pipe materials from corrosive environments so nowadays visco-elastic coatings are best choice for its competitive cost and easier application but since limited studies available in these types of coatings which gives me the chance to better understanding the performance of these coatings in soil simulating conditions.

As a Corrosion Engineer in SWCC-DTRI, I am assigned to evaluate and study the performance of four commercial rehabilitation coating systems noted A, B, C and D electrochemically.

3.2 Objectives

As per agreement with SWCC and coating manufacturer, part of the study will be my master thesis with following objectives:

- Electrochemical investigations of the corrosion performance and ageing of four different commercial thick rehabilitation coatings.

- Determine water absorption behavior and its impact on structure of different commercial thick rehabilitation coatings.

CHAPTER 4

MATERIALS & EXPERIMENTAL PROCEDURES

4.1 Materials:

4.1.1 Materials for Gravimetric Method

Different commercial rehabilitation coatings were received as a roll, Square sample (4*4 cm^2) was cut down from the rolls to evaluate water absorption behavior of four rehabilitation coatings. Two rehabilitation coating categories were studied including low viscosity, low crystallinity (consisting of inner and outer layers) and reinforced polymeric tapes. Three products of low viscosity and low crystallinity were noted B, C and D whereas one reinforced polymeric tape coating was noted A. Two samples were considered for each coating system to ensure reproducibility.

4.1.2 Materials for Electrochemical Study

The pipeline material for Riyadh water transmission is low carbon steel Grade X-52 & X-60. In the present work, X52 was used in the form of spool of pipe with 8" diameter and 0.5 m length. The chemical composition of this grade was analyzed by Optical Emission Spectroscopy (OES) & Carbon Sulfur Analyzer (Table 4.1).

Table 4. 1 Chemical composition of tested Steel pipe.

Element	Fe	C	Mn	Si	Cr	Ni	Mo
Pipe sample	Bal.	0.05	0.84	0.23	0.015	0.13	0.006
Typical composition (max.)	Bal.	0.16	1.6	0.45	0.2	0.2	0.08

The pipe was purchased and cut into required dimensions then sand blasted using portable CLEMCO abrasive blast machine shown in (Figure 4.1). Coating application was assigned to each coating manufacturer inner & outer layers as shown in Figure 4.2).

Figure 4. 1 Portable CLEMCO abrasive blast machine & tested steel pipe before application of coating.

Figure 4. 2 Tested pipe after application of inner & outer layers of the coating.

Each coated spool of pipe contain three cells with defined surface area exposed to the soil simulating solution as shown in Figure 4.3. For viscoelastic coatings, and to explore the impact of coating thickness, one of the cells will be only one layer which is responsible for corrosion protection and the others will be complete system (inner and outer layers) for electrochemical tests. Due to high thickness of coatings it was necessary to examine high surface area of the pipe in order to get response of the coatings. The cell surface area was 78.5 cm2.

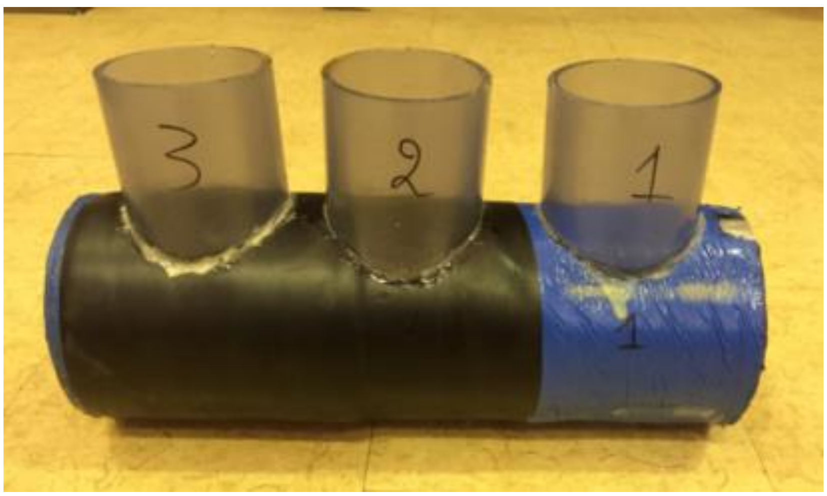

Figure 4. 3 Tested Pipe after installation of the cells.

4.2 Electrolyte/Solution Preparation:

4.2.1 Solution for Gravimetric Method

Two different solutions (Distillate water & 1% NaCl) at two different temperature (ambient & 30 °C) were used to study the water absorption, the tests were conducted for a period of 100 days.

4.2.2 Solution for Electrochemical Study

A soil simulating solution named NS1 was used for chemical ageing of coated samples for more than 200 days. Chemical composition of the NS1 solution as per previous works [49] is shown in Table 4.2 which prepared using analytical grade and deionized water.

Table 4. 2 Simulated soil solution (NS1) chemical composition.

Component	KCl	$NaHCO_3$	$CaCl_2.2H_2O$	$MgSO_4.7H_2O$
Composition (g/l)	0.149	0.504	0.159	0.106

4.3 EXPERIMENTAL TECHNIQUES

4.3.1 Water Absorption by Gravimetric Method.

Water absorption by gravimetric method was conducted for square coating samples immersed in distillate water & 1%NaCl solution at two different temperatures (ambient & 30ºC). Analytical balance was used for weighing coating samples for a duration up to 100 days. For two layer coatings, a defined load was applied prior to immersion test on square samples to minimize microscopic gap that could exist between layers. Figure 4.4 shows schematic representation of the experiment.

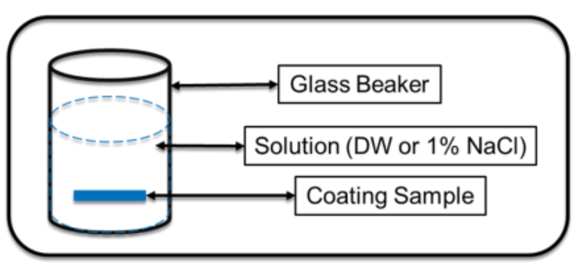

Figure 4. 4 Schematic representation of experimental Set-up.

The increased in weight was calculated based on the following formula according to test standard ASTM-570 [36]:

Increase in weight (%) = [(Wet-conditioned)/conditioned] *100

4.3.1.1 Fourier Transform Infrared Spectroscopy (FT-IR) Characterization

ATR/FTIR analyses were performed on fresh samples (unexposed) as well as an aged or exposed samples to water absorption test. Spectrum one Perkin-Elmer spectrometer was used which contained a ZnSe flat plate crystal at a nominal incident angle of 45°, and an average depth penetration of 2 μm and 16 accumulations with a resolution of 4cm-1 was used. The same force was implemented in all coating samples. Nitrogen was used for purging to avoid interference with atmospheric moisture. Spectra were corrected to take into account the wavelength dependence of ATR and baseline corrected.

4.3.2 Electrochemical Investigations

Autolab Potentiostat model PGSTAT302N is used to carry out the electrochemical measurements as (Figure 4.5); the pipe material substrate will be used as working electrode, graphite electrode as a counter and the Ag/AgCl as reference electrode as shown schematically in Figure 4.6. AC signal with amplitude of 10 mV was applied on coated spool of pipes at open circuit potential (OCP) for a frequency ranging from 10 kHz to 0.01 Hz. The tests were conducted for a period of more than 200 days at an ambient temperature. The experiment was started with measuring OCP for a half an hour then bode & phase modulus was draw to extract impedance & phase angle at specific frequency. All experiments were measured twice a week for each coating type.

Figure 4. 5 Electrochemical Potentiostat.

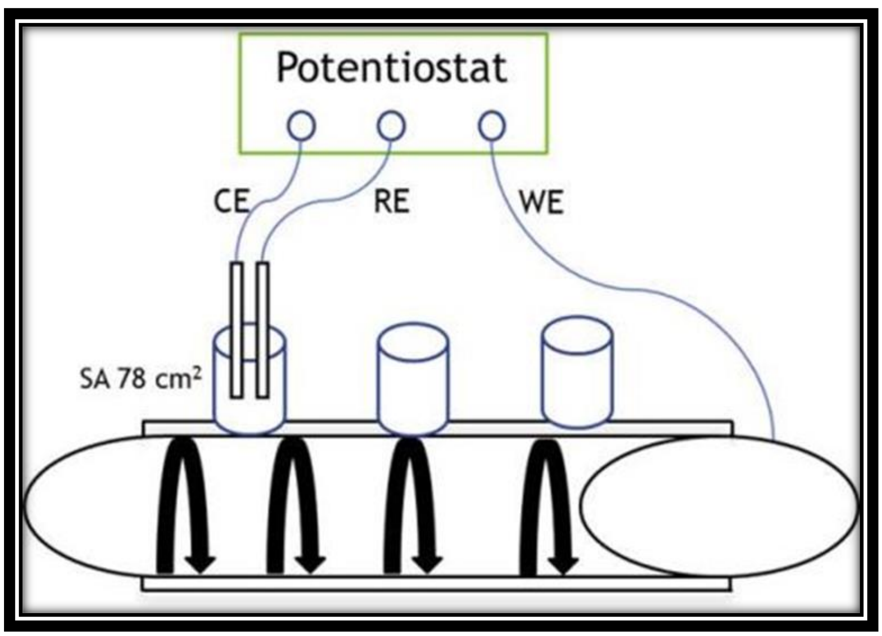

Figure 4. 6 Experiment set-up of electrochemical tests.

CHAPTER 5

RESULTS & DISCUSSION

5.1 Water Absorption by Gravimetric method

5.1.1 Water absorption monitoring without outer layer

5.1.1.1 Distillate water

Figure 5.1 shows the comparison of water uptake behavior of four different rehabilitation coatings immersed in distillate water at room temperature (left side) & 30 °C (right side) respectively, as it can be seen clearly, two permeability's behavior observed, initially there was slight increase in the weight noticed in all coatings with fairly stable value of water volume in products B&C , while product D showed linear increase with time, However product A showed highest permeability's from early stage of immersion which may correlate with compatibility of this product with Cathodic Protection (CP). Products B & C obey the same trend with lowest values among others indicate the high isolation of these coatings while product D showed a typical sigmoidal absorption curve or S-shaped while Fickian's or two stages absorption noticed in product A [36]. In addition, when the temperature increase to 30 °C the behavior of the rehabilitation coatings remain the same with slight increase in diffusion characteristics especially noted in product D.

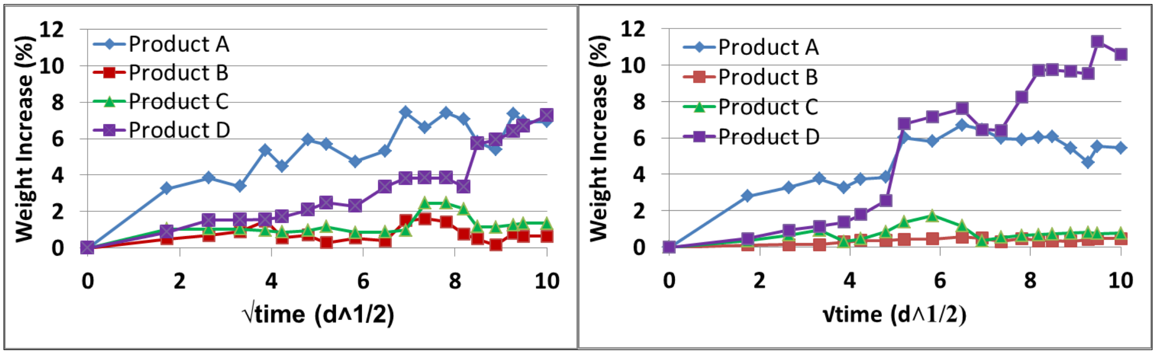

Figure 5. 1 Comparison of Coatings Inner layer Water Absorption immersed in distillate water left side) room temperature & right side) 30°C.

5.1.1.2 Sodium Chloride (1% NaCl)

Figure 5.2 shows the behavior of four thick rehabilitation coatings immersed in 1%NaCl at room temperature (left side) and 30°C (right side), product A showed sharp increase in water absorption specially at room temperature then slight decrease which may refer to pores block by salts while product D showed linear increase in water absorption in both temperatures, however, products B & C showed low level of water absorption.

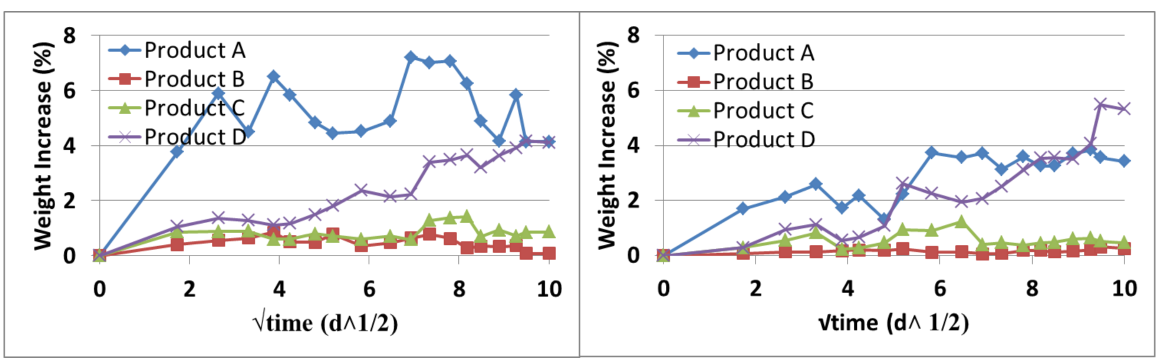

Figure 5. 2 Comparison of Coatings Inner layer Water Absorption immersed in 1% NaCl, left side) room temperature & right side) 30°C.

5.1.2 Water absorption monitoring with outer layer

5.1.2.1 Distillate water

The water absorption behavior was measured considering total system (Inner & Outer layer); Figure 5.3 shows the weight increase versus square root of time of rehabilitation coatings immersed in distillate water at room temperature (left side) and 30 ˚C (right side). Products C & D showed similar behavior observed at room temperature and product B showed lowest water absorption level, however product D showed highest water absorption when the temperature increased to 30 ˚C.

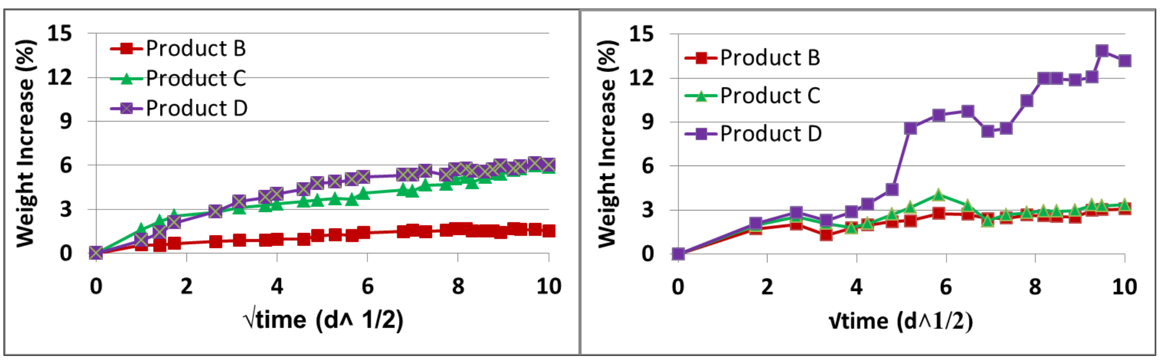

Figure 5. 3 Comparison of Coatings Inner & Outer layer Water Absorption immersed in distillate water left side) room temperature & right side) 30°C.

5.1.2.2 Sodium Chloride (1% NaCl)

Figure 5.4 shows the behavior of rehabilitation coatings immersed in 1%NaCl at room temperature & 30 ˚C respectively. The same trend was observed between DW and 1%NaCl. Product C showed high water absorption at room temperature which may refer to micro gap exists between the layers.

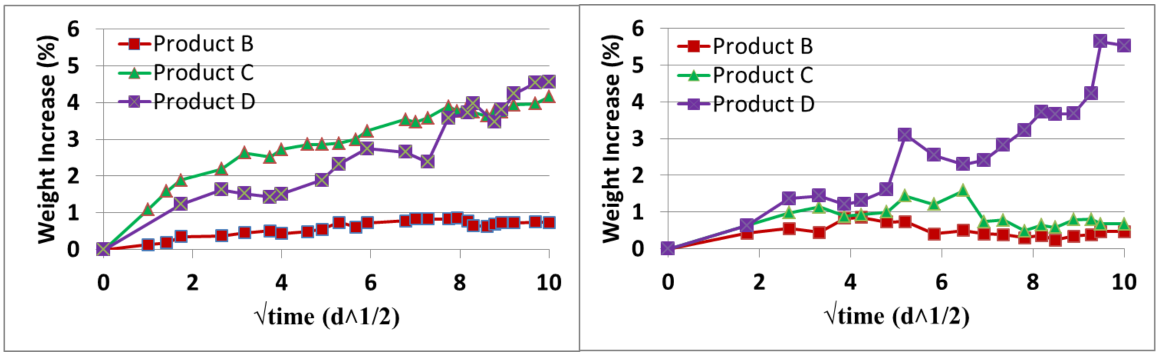

Figure 5. 4 Comparison of Coatings Inner & Outer layer Water Absorption immersed in 1% NaCl, left side) room temperature & right side) 30°C.

Based on the above results ,the below table summarizes the conditions and rank the four rehabilitation coatings ,1 indicates the highest water absorption and 4 is the lowest water absorption, almost product D has the highest values in all conditions and product B is the lowest in all conditions except distillate water at room temperature.

Table 5. 1 Rehabilitation coatings ranking in different environments.

Property	Product A	Product B	Product C	Product D
Water absorption (DW Room Temp.)	4	3	2	1
Water absorption (DW 30 °C Temp.)	2	4	3	1
Water absorption (1%NaCl Room Temp.)	2	4	3	1
Water absorption (1%NaCl Room °C Temp.)	2	4	3	1

5.1.3 ATR/FTIR Characterization

5.1.2.1 Fresh Samples:

FT-IR spectroscopy can be used in an identifications of the coating type (pigments, cure time, contamination , degradation …etc.), D.Banerjee et al.[37] carried study on different organic and inorganic coatings to evaluate them after salt spray test, then FT-IR spectra were collected to compare with fresh samples, the below table shows the summery of the findings:

Table 5. 2 FT-IR comments on different organic and nonorganic coatings [37].

Coating System	FT-IR Comments	Notes
Coal Tar Epoxy	No much difference in transmittance between aged and un-aged samples.	Resin not degrade in the samples subjected to salt spry
Glass Flake Epoxy	No much difference in transmittance between aged and un-aged samples.	No degradation.
Polyurethanes	Additional vibration bond at 719 cm^{-1} corresponds to C-H vibrational bond.	The additional vibrational bond was due to interaction between coating and salt spry indicates slow corrosion process.
High Build Epoxy	No change in transmittance between aged and non-aged coatings.	Vibration bond at 1219,772 cm^{-1} corresponds to CH symmetry vibration and C-H bending mode with aromatic compound.
Concrete	Single vibration bond at 772 cm^{-1} corresponds to C-H bending mode.	Sharp decrease in transmittance due to forming of new C-H bending mode.

Figure 5.5 shows FT-IR spectrum of fresh rehabilitation coating samples (not exposed) as it can be observed the peaks at 1400 cm-1 indicating CH2 or CH3 bonds which belongs to aliphatic (alkane) group and C-H bond at 2800 cm-1, the products C and D have the same structural components exactly and it differs from others but the stretching of all bonds are at the same wavenumber. Overall the peaks are indicating organic functionality compounds.

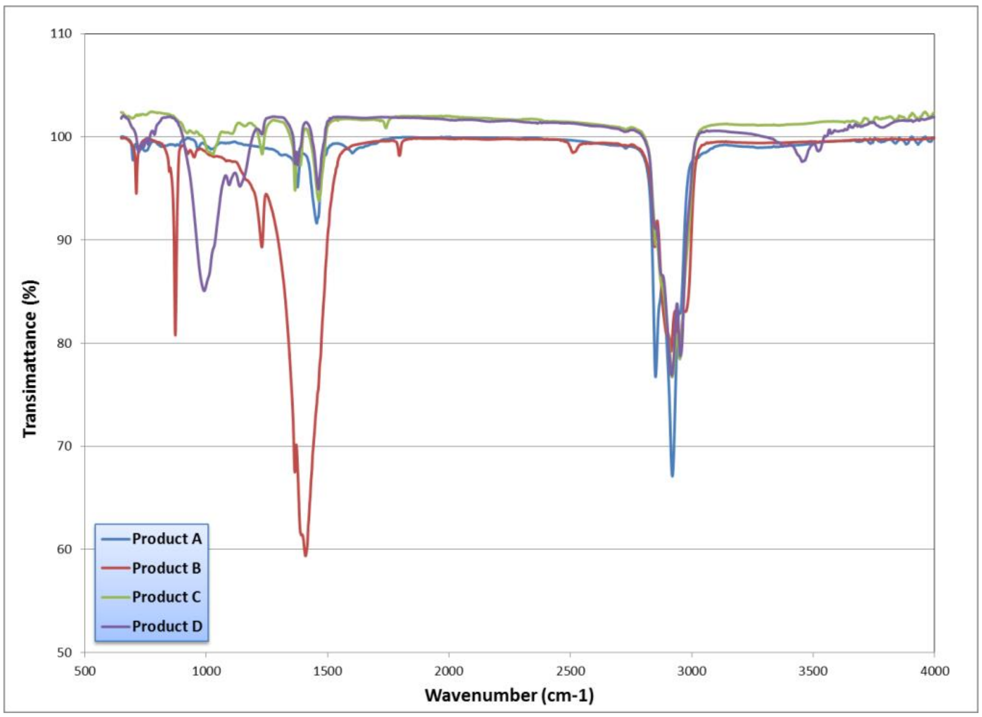

Figure 5. 5 FT-IR spectrum of fresh samples (Not-exposed).

5.1.2.2 Aged Samples:

It was necessary to compare the aged with un-aged samples to see the changes in structural compounds after interaction with different electrolytes and to determine the effects of temperature. Figure 5.6 shows ATR/FTIR analysis of Product A at DW and 1%NaCl at room &30° C temperature as it can be observe from the figures new stretching bands are developed around 3400 cm-1 which is corresponds to hydroxyl stretching & water molecules confirmed the high permeability of this coating , the changes in hydroxyl stretching may due to three reasons :i)Alcohol or hydro peroxide formation ii) Phenol formation and iii) water trapped in alkyd and the changes from 1600 to 1650 cm-1 mainly due to water trapped (OH stretching as well as accumulations of vinyl groups. [38]. Another observation could be noticed at the main stretching band around 2900 cm-1, the intensity of the aged samples start to decrease which may disappear after some time as it can be seen in the bands around 700 cm-1 some of the bands are completely destroyed in 1% NaCl & DW at 30°C.

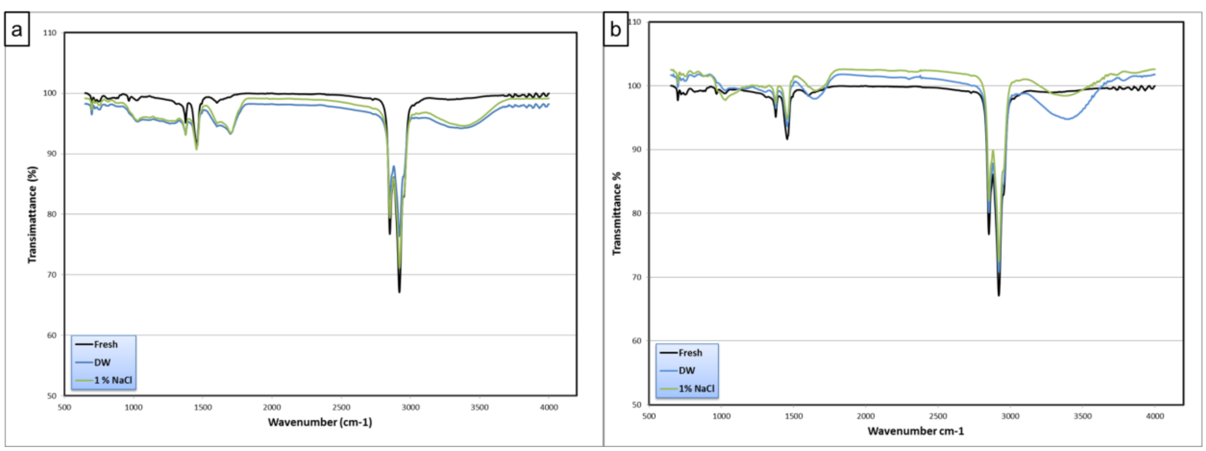

Figure 5. 6 FT-IR spectrum of Product A. a) Room Temperature & b) 30 °C Temperature.

Figure 5.7 shows the ATR/FTIR spectrums of the product B in room temperature, No stretching band at 3400 cm-1 indicates the impermeable coating and absence of hydroxyl molecules while there was main stretching band observed around 1400 cm-1 which may refer to reorganizing of polymer chain to block the pores.

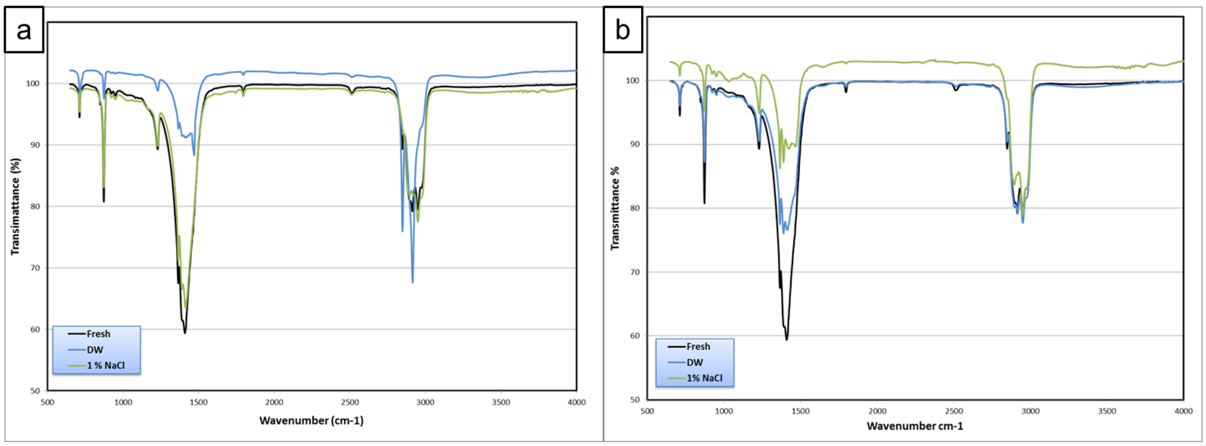

Figure 5. 7 FT-IR spectrum of Product B. a) Room Temperature & b) 30 °C Temperature.

Product C in figure 5.8 shows the same behavior of product B but weak peaks are their at 3700 cm-1 indicating little absorption in DW only ,the rest are the same and no structural changes are there.

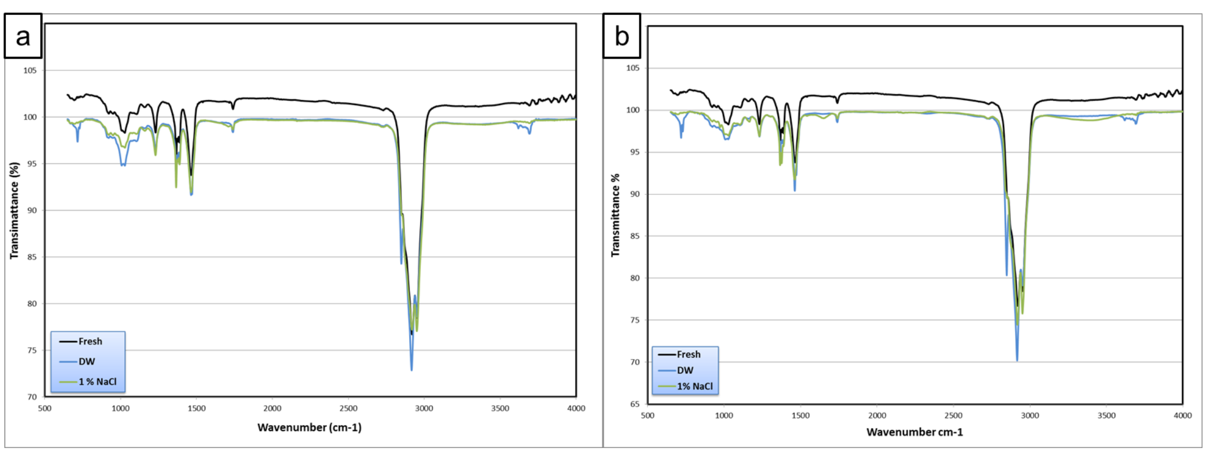

Figure 5. 8 FT-IR spectrum of Product C. a) Room Temperature & b) 30 °C Temperature.

Figure 5.9 shows product D at room temperature & 30°C temperature respectively, as it is clearly shown no any changes in structural compounds are observed indicates the high permeability without any negative effects on the structure.

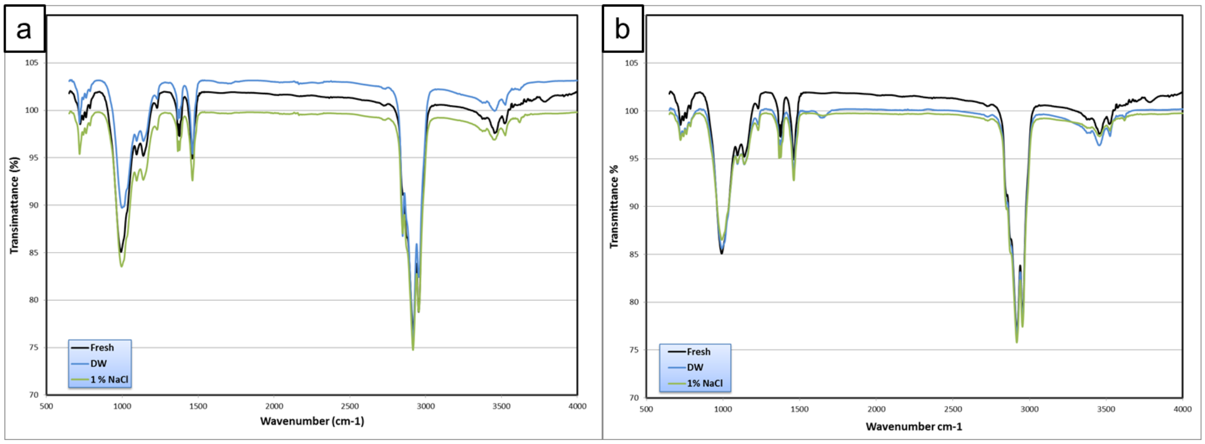

Figure 5. 9 FT-IR spectrum of Product D. a) Room Temperature & b) 30 °C Temperature.

5.2 Electrochemical Investigations Results

5.2.1. Open Circuit Potential Monitoring:

The monitoring of Open Circuit Potential (OCP) is well established as one of the non-destructive electrochemical measurement giving indication on the different processes occurring at the metal surface. Applied for either coated or un-coated metal substrate, OCP values can give indication on the corrosion initiation, passivation phenomenon or cathodic protection process [9, 39]. In the present study, the metal substrate is coated with significantly thick (in the order of millimeters) coating where OCP measurement is quite difficult. Indeed, the measurement is difficult due to the low progress of the electrolyte particularly related to the high thickness of coating structure, and the high electrical resistance of the coating.

Figure 5.10 shows OCP values for the different studied coatings applied on X52 steel. In addition to bituminous product, the results include low viscosity coatings with and without outer layer (PVC). The test was conducted for duration more than 250 days in soil simulating solution NS1. The OCP was also measured for the uncoated X52 steel showing a value of -0.8 V/Ag/AgCl. For the coated samples without PVC, we distinguish two stages where the first one duration was up to 13 weeks with highly fluctuating positive potentials. The second phase shows a decrease of potentials toward negative values approaching the corrosion potential of the uncoated steel substrate where the four coatings behavior is split into two groups. Product A & D showed similar rapid decrease with potentials approaching the X52 steel substrate. However, Product B & C present less negative potentials which indicate slow progress of solution to the metal substrate. In the presence of the outer PVC layer, the same trend was observed a curious rapid decrease of potential values for the product D coated steel started after 3 weeks of immersion. Considering the high thickness with outer PVC layer (6.4 mm), this result could be due a corrosion initiated before the application of the coating.

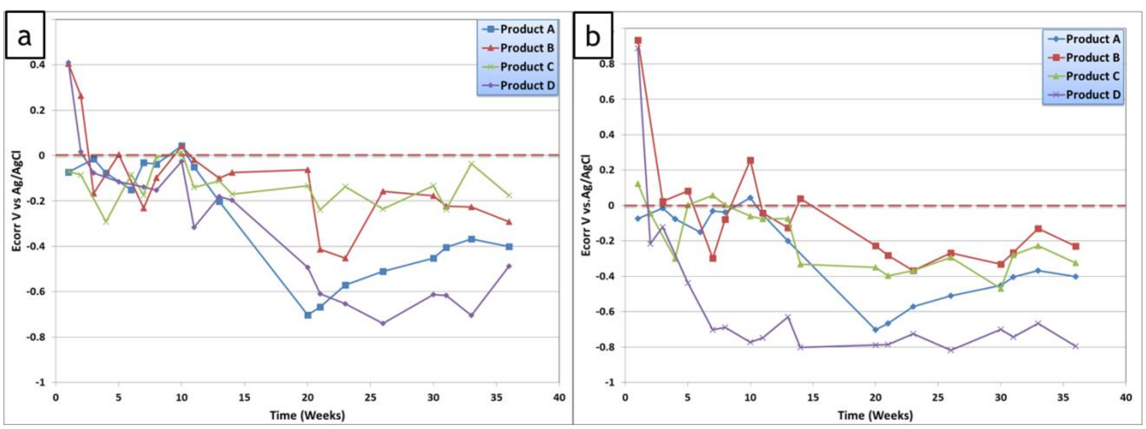

Figure 5. 10 Ecorr evolution of different commercial rehabilitation coatings a) without PVC and b) with PVC.

5.2.2 Electrochemical Impedance Spectroscopy (EIS) Monitoring:

EIS is a powerful tool to assess the ageing of coatings. Typical EIS response for low viscosity coatings showed one time constant with straight line in Nyquist diagram and a phase angle shift equal to 90°. Usually, the monitoring of the impedance magnitude at 0.1 Hz during long immersion duration give good indication on the ageing and barrier properties of the coating against electrolyte ingress. An excellent coating should provide a magnitude higher than 10^9 $\Omega.cm^2$ [35, 40]. Figure 5.11 show impedance magnitude for both inner and total coating system of products B, C and D compared with product A (one layer). In both situations, the criterion was satisfied up to 13 weeks of immersion. After that, product A& D showed a rapid decline in this magnitude indicating the starting of corrosion at the interface metal/inner coating layer. For product B&C, the impedance decline start to be visible after 27 weeks of immersion.

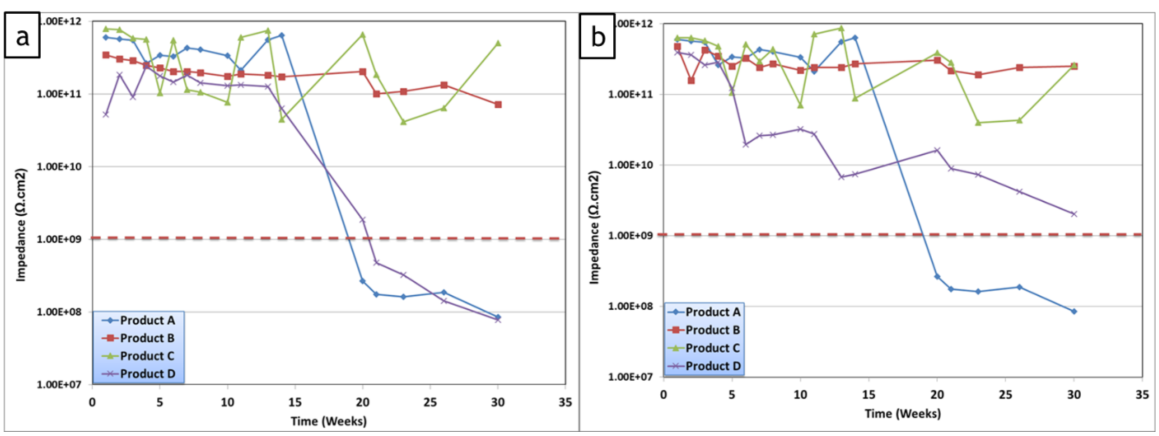

Figure 5. 11 Evolution of Impedance magnitude at 0.1 Hz for different commercial rehabilitation coatings in soil simulating solution a) without PVC & b) with PVC.

Such impedance magnitude decline was also observed in the case of the total coating system (inner & outer layers). This result is in agreement with OCP monitoring shown in Figure 5.10 as well as with phase angle shift plot at 1 kHz in Figure 5.12. In fact, a capacitive behavior (blocking electrode) appears in the phase angle plot at higher frequencies with values close to 90°.

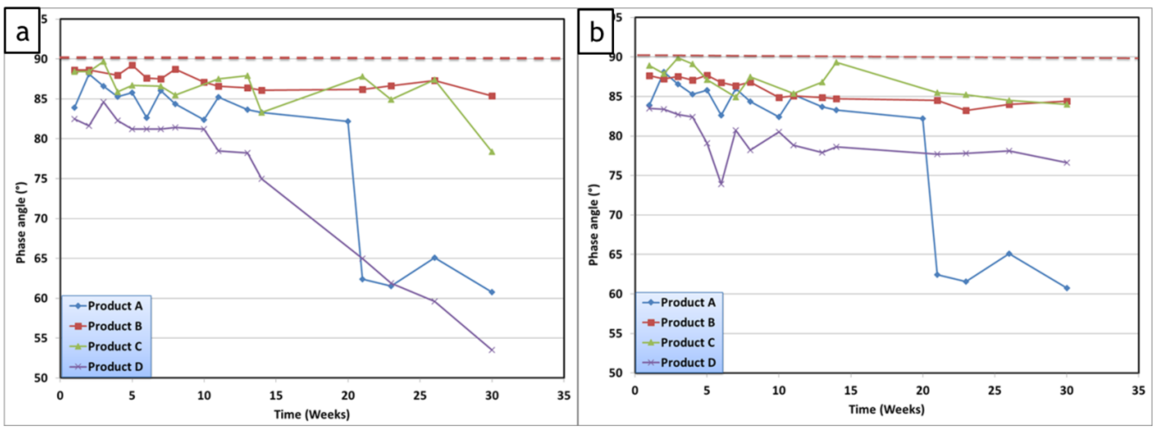

Figure 5. 12 Evolution of Phase angle at 10 kHz for different commercial rehabilitation coatings in soil simulating solution.

As per ISO 21809 the specific electrical resistance should be $\geq 10^8$ Ω after immersion in 1% NaCl at room temperature in non-crystalline low viscosity polyolefin based coating [41]. The change of bode plot slop and appearance of second time constant indicates that the water reached to the substrate and this may due to adhesion deterioration of the coating [42].

Figures 5.13 – 5.16 show bode plot at 1,100,150 &230 days for candidates coating products , all the coating systems have one capacitive behavior at the beginning of the tests specially at 1 day indicating the high isolation of the coating barriers to the electrolyte, product A (one layer only) in figure 5.13 shows one capacitive behavior until 150 days then at 230 days the impedance significantly decreased at low frequency region indicates new resistive properties or corrosion process at metal /coating interface. The decrease in the low frequency impedance indicates initiation of the corrosion process of the substrate as well as the diffusion characteristics of the coating. The fluctuation in the curves noted in the middle region of the frequency (1-100 HZ) represents the relaxation process and does not affect the evaluation of the coating performance.

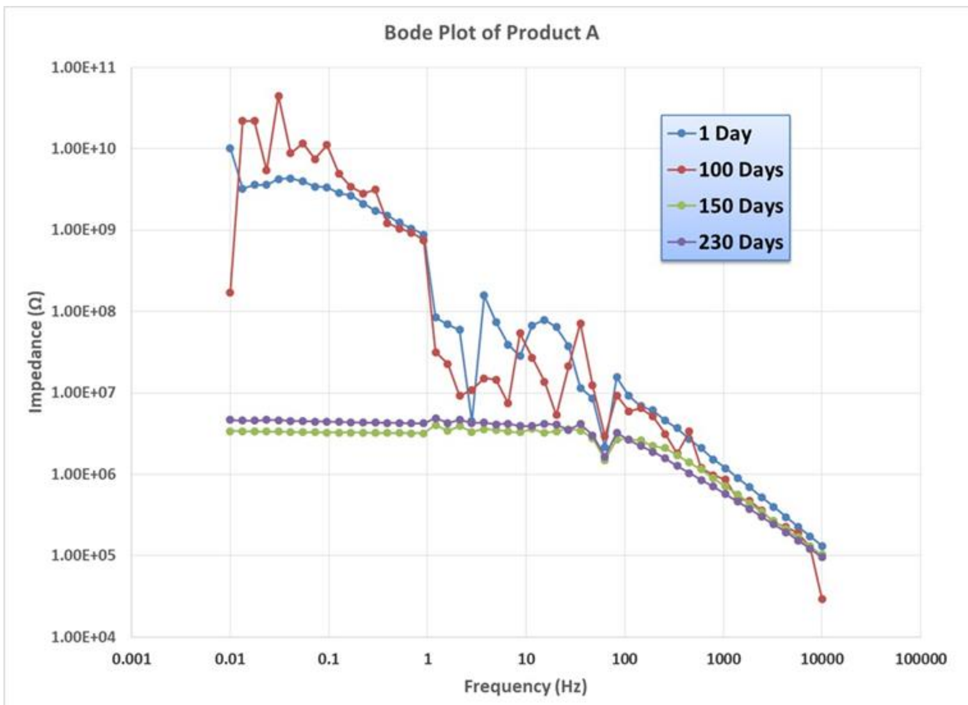

Figure 5. 13 Bode Plot for different immersion times 1,100,150 &230 days of product A.

Figure 5.14 represents the bode plot of product B with and without outer layer PVC, this was done to simulate the worth condition in case of improper application of outer layer, as it can be noticed the decrease in an impedance values at low frequency region was gradually up to 150 days then significant decrease observed after 230 days. However, in presence of outer layer PVC, the one capacitive behavior is dominant with different exposure time indicates outstanding performance of this system.

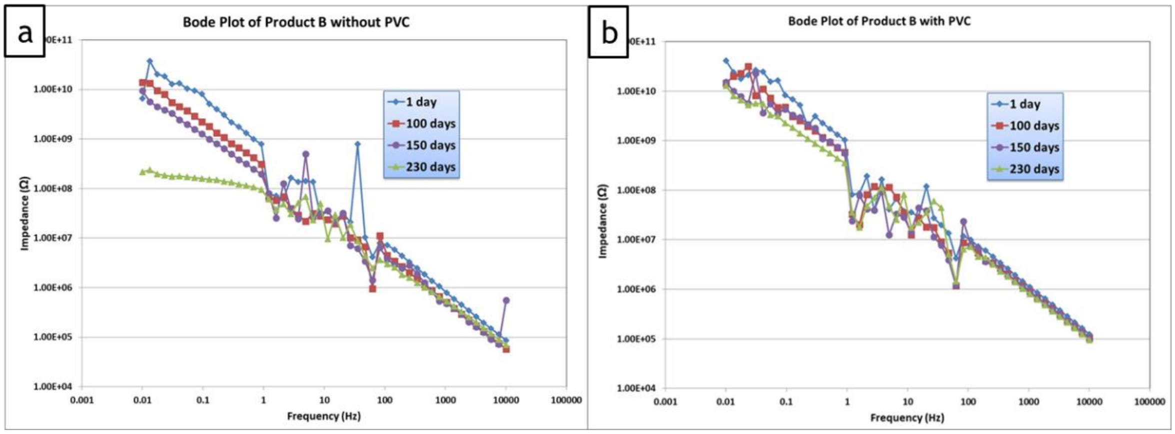

Figure 5. 14 Bode Plot for different immersion times 1,100,150 &230 days of product B, a) without PVC and b) with PVC.

Figure 5.15 shows product C without PVC at different times, in the beginning of immersion higher impedance could be observed then it decreased after 100 days after that again it increase to high value indicates reorganized of the barrier properties or prevention against deterioration ,the same behavior noticed in presence of outer layer PVC.

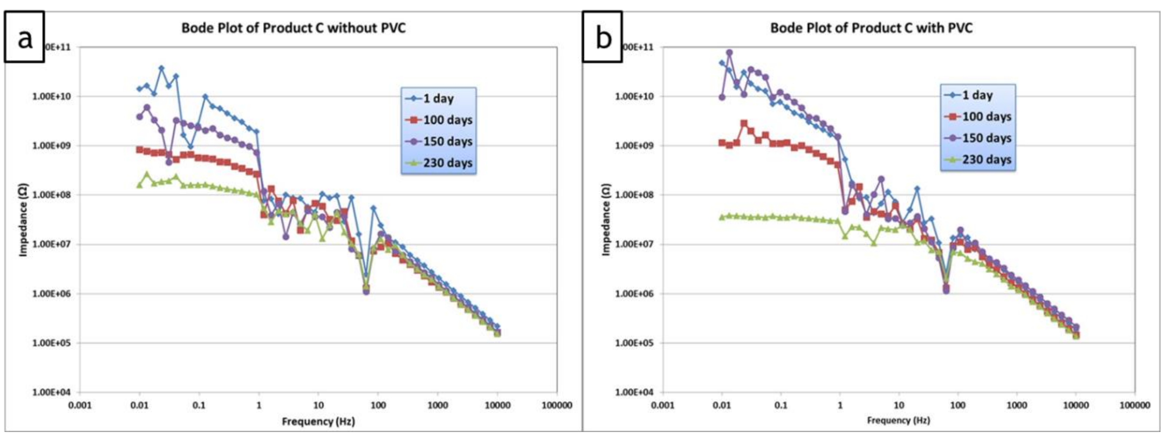

Figure 5. 15 Bode Plot for different immersion times 1,100,150 &230 days of product C, a) without PVC and b) with PVC.

Figure 5.16 shows the bode plot of product D without PVC, the deterioration or drop in an impedance is gradually at low frequency region, Moreover the deterioration of product D with PVC was noticed in earlier stage of immersion Fig.5.20 and this may refer to improper application.

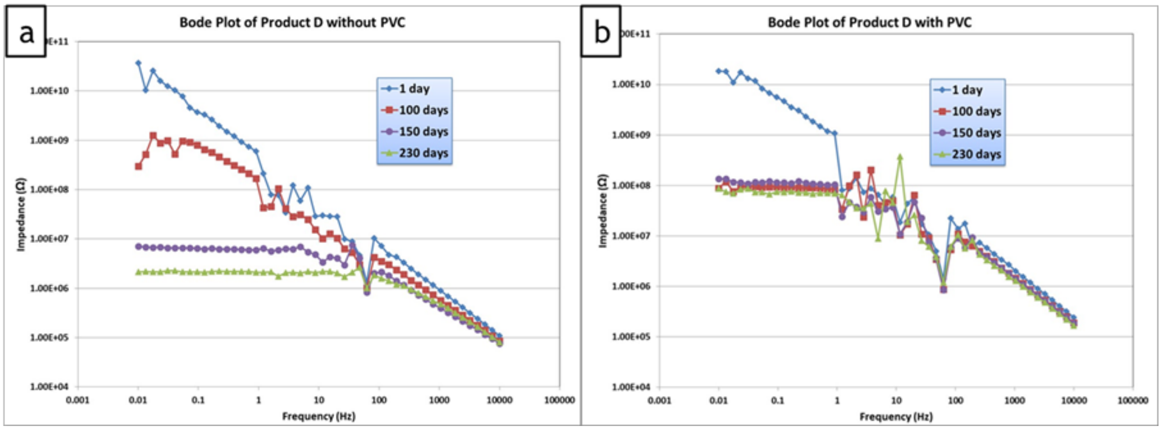

Figure 5. 16 Bode Plot for different immersion times 1,100,150 &230 days of product D, a) without PVC and b) with PVC.

CHAPTER 6

CONCLUSIONS & RECOMMENDATIONS

6.1 Conclusions

Four types of coating products were proposed for rehabilitation of underground pipeline. Electrochemical tests were carried out in presence and absence of outer layer PVC, these tests include: OCP and EIS monitoring in order to evaluate and rank the performance of the rehabilitation coatings. Overall the results obtained indicate the slow progress of the electrolyte into the substrate. All the coatings are having one time capacitive behavior in the initial stages of immersion and this may refer to high thickness of these coatings. However, the highest thickness coatings should not be considered as the lowest water absorption and vice versa. The application of the outer layer play an important role since the deterioration of the products without outer layer is more rapid than the products with outer layer. Coating A & coating D without PVC are having the same behavior deteriorated in the same manner but when the outer layer PVC was added to coating D the performance of the coating improved and the impedance at the end of exposure was higher than the impedance without PVC. Coating B with & without PVC showed outstanding performance and stability during exposure with little deterioration in the absence of outer layer PVC. Coating C exhibited similar trend compared to coating B with some fluctuations on the behavior indicating the difficulties of the electrolyte ingress, however the performance was good and the deterioration was observed after 30 weeks. The application of the outer layer PVC is very important and it reflects very high impact on the behavior of the coating.

Overall performance of the tested coatings in terms of corrosion protection could be ranked without considering cathodic protection from the lowest to highest absorption as coating B with PVC, coating B without PVC, coating C with PVC, coating C without PVC, coating D with PVC, coating A and coating D without PVC.

Based on the Gravimetric method findings the conclusions:

- Product D has the highest absorption in DW at room temperature and product A has the lowest value, products B & C show the same trends.
- Product D and A have high water absorption in 1%NaCl at room temperature approximately 4-5 % and products B & C approximately have 0.5-1.2 % water absorption after full drying.
- In DW at 30 °C the water absorption of product D increase significantly after 23 days of immersion reaching 12 % after 100 days.
- The behavior of the coatings in 1%NaCl at 30 °C is the same as at room temperature.
- ATR/FTIR spectrums indicate the presence of alcohol peaks in products A & D.
- The coating products could be ranked as the following from the lowest water absorption to highest (B-C-A-D).

6.2 Recommendations

The current aim of thesis work was to rank and evaluate the corrosion protection performance of different coatings proposed for rehabilitation project without talking into account the combination of these coatings with cathodic protection (CP). Further tests are required in order to investigate the coatings performance when they combine with CP. The following points need to be considered in order to investigate such type of coatings:

- Longer time of monitoring is required to investigate the water uptake through EIS technique.
- Cathodic disbonding (CD) is the only test that can be used to determine whether these coatings are compatible with CP or not.
- Soil Stress test need to be carried out to investigate the ability of the coating to resist mechanical damage.
- The decision of the coating selection should be carried out based on laboratory results as well as field trial.

References

[1] Standard Practice "Control of External Corrosion on Underground or Sumerged Metallic Piping Systems" SP 0169, 2013 NACE International, item no.21001.

[2] John A. Beavers and Neil G. Thompson "External Corrosion of Oil and Natural Gas Pipelines" ASM International,volume 13C, 2006.

[3] Kenneth B.Tator "Laboratory Testing of Pipeline Exterior Coatings- a commentary on correlation with field performance." NACE International, 2006 paper no.06042.

[4] NACE Standard "Control of External Corrosion on Underground or Submersed Metallic Piping Systems" SP0169-2013, Item no.21001.

[5] JF.Doddema Clever Coatings, "the uses of self-healing visco-elastic coatings on underground pipelines" World Pipelines, October, 2007.

[6] Richard norsworthy "Understanding corrosion in underground pipelines: basic principles", Technical Report, 2014.

[7] Abboud L. Mamish "Tape Coating System for Pipeline Corrosion Protection System".

[8] A.W.PEABODY "CONTROL OF PIPELINE CORROSION",Second Edition,NACE International,2001.

[9] Fu, A.Q., and Y.F. Cheng. "Characterization of the permeability of a high performance composite coating to cathodic protection and its implications on pipeline integrity." Organic Coating, pp 423-428,2011.

[10] Lee, S.H., W.K. Oh, and J.G. Kim. "Acceleration and quantitative evaluation of degradation for corrosion protective coatings on buried pipeline: Part II. Application to the evaluation of polyethylene and coal-tar enamel coatings." Organic Coating, pp 784-789,2013.

[11] Ali N Moosavi "Advances in Field Joints Coatings for Underground Pipelines" NACE International, 2000 paper no.00754.

[12] Chris Alliston, Banach and Joe Dzatko , Liquid Skin, "The development of liquid epoxy coatings for extending the life of existing pipelines" World Pipelines, November, 2002.

[13] Michael Schad "Rehabilitation of corrosion protective coatings on buried steel pipelines" Technical Report, Corrosion Protection, 2012.

[14] Richard norsworthy "Coatings used in conjunction with cathodic protection – shielding vs. non shielding pipeline coatings" NACE International, 2009 paper no.4017.

[15] NACE standard SP0109 "Field Application of Bonded Tape Coatings for External Repair, Rehabilitation, and Weld Joints on Buried Metal Pipelines" NACE International, 2009 Item no.21143.

[16] Dennis Neal "Pipelines Coatings Failure –Not only what you think it is" NACE International, 2000 paper no.00755.

[17] Richard Norsworthy and Chic Hughes "Proven protection" World Pipelines, Coatings & Linings, November, 2007.

[18] Roland Palmer-Jones, Phil Hopkins, David Eyre "Pipeline rehabilitation planning "Rio Pipelines, Technical Paper, 2005.

[19] International Standard ISO/FDIS 21809-3:2015 "Petroleum and natural gas industries. External coatings for buried or submerged pipelines used in pipeline transportation systems. Part 3: Field joint coatings".

[20] Drs JF Doddema "the use of visco-elastic self-healing pipeline coating" NACE International Corrosion, 2010, paper no.10042.

[21] Rischard Norsworthy "Addresing soil stresses and CP shielding bu using a woven geotextile fabric backed tape system with rubber modified bituminous compound" NACE International Corrosion, 2000, paper no.00769.

[22] Moufaq I. Jafara, Fikry F. Barouky and Khalid Al-Mugary "Evaluation of Visco-elastic Coatings – A User Perspective" Saudi Aramco, Technical Report.

[23] Michael Romano , Matt Dabiri and Alan Kehr "Protecting and Maintaining Transmission Pipeline" A JPCL eBook, 2012.

[24] A. F. Baldissera, D. B. Freitas and C. A. Ferreira"Electrochemical impedance spectroscopy investigation of chlorinated rubber-based coatings containing polyaniline as anticorrosion agent" Materials and Corrosion,Vol.61,No.9, PP: 790-801 ,2010.

[25] E.M.Fayyad, M.A. "Evaluation Techniques for the Corrosion Resistance of Self-Healing Coating" Electrochemical Science, 2014.

[26] Marie-Georges Olivier and Mireille Poelman "Use of Electrochemical Impedance Spectroscopy (EIS) for Evaluation of Electrocoatings Performance" Recent Researches in Corrosion Evaluation and Protection, 2012.

[27] Hensley, J.R. Scully and S.T. "Lifetime Prediction for Organic Coatings on Steel and a Magnesium Alloy Using Electrochemical Impedance Methods" NACE International, 1994.

[28] F.Deflorian and I.Felhosi,"Electrochemical Impedance Study of Environmentally Friendly Pigments in Organic Coatings" NACE International, February, 2003 .

[29] Gibitta Giovanaa , Massimo Soprani and Antonio Bennardo "Assessment of a circuital model for EIS measurements on buried pipelines" NACE International 2000 paper no.00765.

[30] E.McCafferty. Introduction to corrosion science. Washington: Springer science+Business media, 2009.

[31] F. Mansfeld and C.H Tsai "Determination of Coating Deterioration with EIS I.basic relationships." National Association of Corrosion Engineers,December 1991.

[32] X.M.Li, B.Faber,B.Mirch ,and H.Castaneda "Analysis of Soft Coating Corrosion Performance on Carbon Steel Using Electrochemical Impedance Spectroscopy" NACE International, Vol.70,No.6, 2014.

[33] B. Liu, Y. Li, H. Lin,and C.-N. Cao "Electrochemical Impedance Spectroscopy Study on the Diffusion Behavior of Water through Epoxy Coatings" NACE International, Corrosion_Vol.59,No.9, 2003.

[34] Hung.M Ha & Akram Alfantazi "Water Transport in Polymer Coatings and the correlation between coatings properties and water uptake in aquios solutions at elevated temperttures " CORROSION_Vol.72,No.1, January 2016.

[35] Y. Gonza´lez-Garcı ´ a, S. Gonza´lez, R.M. Souto "Electrochemical and structural properties of a polyurethane coating on steel substrates for corrosion protection" Corrosion Science 49, pp:3514-3526, May 2007.

[36] G.K.van der Wel , O.C.G.Adan "Moisture transport and equilibrium in organic coatings" HERON, Vol.45,No.2 ISSN 0046-7316 ,2000.

[37] D.Banerjee, C.K.Ghosh, B.Duari, R.Dey, B.Chaudhuri "Failure analysis of resin of different coating materials by infrared spectroscopy" Volume 3, Issue 3: Page No. 105-108, May-June 2014.

[38] F.Perrin, C.Merlatti, E.Aragon, A. Margaillan" Degradation study of polymer coating: Improvement in coating weatherability testing and coating failure prediction" Organic Coatings 64 (2009) 466–473.

[39] N. Kouloumbi, G.M. Tsangaris, A. Skordos, S. Kyvelidis "Evaluation of the behaviour of particulate polymeric coatings in a corrosive environment.Influence of the concentration of metal particles " Organic Coatings 28 pp:117-124, 1996.

[40] Philip A.Schweitzer, P>E>. Analytical Methods in Corrosion Science and Engineering. CRC Press, 2006.

[41] International Standard ISO/FDIS 21809-3:2015 "Petroleum and natural gas industries. External coatings for buried or submerged pipelines used in pipeline transportation systems. Part 3: Field joint coatings"

[42] Marie-Georges Olivier and Mireille Poelman "Use of Electrochemical Impedance Spectroscopy (EIS) for the Evaluation of Electro coatings Performances", Recent Researches in Corrosion Evaluation and Protection,2012.

[43] Michea Romano, Matt Dabiri & Alan Kehr "Coatings used to protect oil and and gas pipelines " JPCL, 2005.

[44] Ken Holyoake and Jiangnan Yuan "Electrochemical Impedance Spectroscopy measurements of barrier coatings" Technical Report,Materials Performance Technologies Ltd,1999.

[45] Gordon Bierwagen, Dennis Tallman, Junping Li, Lingyun Hea, Carol Jeffcoate "EIS studies of coated metals in accelerated exposure" Organic Coatings 46,pp: 148–157,2003.

[46] P. L. BONORA,F . DEFLORIANa, nd L. FEDRIZZI "Electrochemical Impedance Spectroscopy as a tool for investigation under paint corrosion" Electrochimica Acta 41, pp:1073–1082, 1996.

[47] F. Deflorian and I. Felhosi Electrochemical Impedance Study of Environmentally Friendly Pigments in Organic Coatings", NACE International CORROSION _Vol.59,No.2, FEBRUARY 2003.

[48] Subrahmanya Shreepathi, Shrikant M. Naik, Mohan Rao Vattipalli "Water transportation through organic coatings: correlation between electrochemical impedance measurements, gravimetry, and water vapor permeability" Coating Technologies, ACA and OCCA,pp:411-422, 2012.

[49] R. A. de Sena, I. N. Bastos, G. M. Platt, Theoretical and Experimental Aspects of the Corrosivity of Simulated Soil Solutions, ISRN Chemical Engineering (2012), 1-6.

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